

**TECHNICAL REPORT
66-38-FD**

AD 634252

**EDIBLE COATINGS
FOR
DRIED AND COMPACTED FOODS:
PART II**

by
Morton S. Cole

**Archer Daniels Midland Company
Minneapolis, Minnesota**

Contract No: DA19-129-AMC-102(N)

May 1966

**UNITED STATES ARMY
NATICK LABORATORIES
Natick, Massachusetts 01760**



Food Division

FD-48

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED.

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

Citation of trade names in this report does not constitute an official indorsement or approval of the use of such items.

Destroy this report when no longer needed. Do not return it to the originator.

Distribution of this
document is unlimited

AD _____

TECHNICAL REPORT
66-38-FD

EDIBLE COATINGS FOR DRIED AND COMPACTED FOODS:

PART II

by

Morton S. Cole
Archer Daniels Midland Company
Minneapolis, Minnesota

Contract No. DA19-129-AMC-102(N)

Project reference:
7X84-06-033

Series: FD-48

May 1966

Food Division
U.S. ARMY NATICK LABORATORIES
Natick, Massachusetts 01760

FOREWORD

This is the second part of a report dealing with the development and application of edible coatings for protection of dehydrated food against moisture, oxygen, attrition through mechanical forces, and surface growth of mold. The first part, Part I, is directed primarily toward the development of edible coatings and their evaluation for their intended purpose, both as an isolated film and as a coating on representative foods. Part II is primarily concerned with commercially feasible procedures for the application of such films and the evaluation of films as applied to representative foods maintained under severe conditions of both temperature and humidity.

This investigation was performed at the Research Center of the Archer Daniels Midland Company, 10701 South Lyndale Avenue, Minneapolis, Minnesota under contract DA19-129-AMC-102, as supported by funds from the project titled, COMBAT FEEDING SYSTEMS. Technical effort was directed by Dr. Morton S. Cole, Official Investigator. He was assisted by M. M. Hamdy, H. S. White and Nancy Williams. The Project Officer for the U.S. Army Natick Laboratories was Dr. Maxwell Brockmann of the Food Division. Alternate Project Officer was Mr. Philip B. Warnock of the Container Division.

**FERDINAND P. MEHRLICH, PhD.
Director
Food Division**

APPROVED:

**DALE H. SIELING, PhD.
Scientific Director**

**W. M. MANTZ
Colonel, QMC
Commanding**

CONTENTS

	<u>Page</u>
List of Tables	vi
List of Figures	vii
Abstract	viii
Introduction	
Summary and Conclusions	3
Experimental Methods	5
Materials	5
Compression of food bars	6
Preparation of coatings	7
Casting from solvents	7
Formation of films from hot melts	8
Sprayed coatings	8
Application of Coatings to Foods by Commercial Methods	9
The Wurster fluidized bed coating process	9
Curtain coating	10
Fusion of powdered coatings during terminal phase of freeze drying	11
Coating by dipping in dispersions and in hot melts	12
Coating of margarine by dipping in hot melts	12
Physical and Chemical Tests	13
Fragmentation	13
Tensile strength of films	13
Elongation	13
Moisture vapor transmissions	13
Oxygen permeability	13
Equilibrium relative humidity	14
Moisture analysis	15
Peroxide value	15
Total carbonyl	15
Organoleptic evaluation	16
Film thickness	16
Continuity of coatings	16
Storage Conditions	16
Treatment of Freeze-Dried Meat with Polar Gases	17

CONTENTS (Con't)

	<u>Page</u>
Results and Discussion	18
Properties of moisture barrier films	18
Properties of oxygen barrier films	19
Application of coatings to freeze-dried and compacted foods	21
Fluidized bed coating	21
Curtain coating	21
Dip coating	23
Coating of margarine	24
Coating during the terminal stage of freeze drying	26
The effect of gases on moisture sorption by freeze-dried chicken	28
Storage studies on coated food bars	30

LIST OF TABLES

	<u>Page</u>
1. Compression of Food Blocks	34
2. Reconstitution of Compressed Food Bars	35
3. Characteristics of Moisture Barrier Films	36
4. Characteristics of Oxygen Barrier Films	37
5. Moisture Uptake by Sodium Caseinate Film at Various Relative Humidities	38
6. Sorption of Gases by Cooked, Freeze-Dried Chicken	39
7. Moisture Sorption by Cooked, Freeze-Dried Chicken Treated with Polar and Non-Polar Gases	40
8. Storage Studies on Food Bars at 40° C, 48% RH	41
9. Oxidative Stability of Food Bars Stored at 40° C, 48% RH	42
10. Storage Studies on Food Bars under Cycling Temperature and Humidity Conditions	43

LIST OF FIGURES

	<u>Page</u>
1. The Eastoflow Machine	44
2. Oxygen Permeability of Sodium Caseinate Films	45
3. Equilibrium Relative Humidity of Sodium Caseinate Film	46
4. Oxygen Permeability of Ethylcellulose-Acetoglyceride-Calcium Stearate Film	47
5. Oxygen Permeability of Ethylcellulose-Amylose Laurate and Ethylcellulose-Calcium Stearate Films	48
6. Moisture Sorption at 50% RH by Freeze-Dried Beef Coated During Freeze Drying	49
7. Moisture Sorption at 75% RH by Freeze-Dried Beef Coated During Freeze Drying	50
8. Moisture Sorption at 98% RH by Freeze-Dried Beef Coated During Freeze Drying	51
9. Moisture Sorption by Gas-Treated Cooked Freeze-Dried Chicken	52
10. Peeling of Coated Food Bars under Cycled Temperature and Humidity Conditions	53
11. Coated Food Bars	54

ABSTRACT

Edible thermoplastic coatings were applied as moisture-oxygen barriers to dry bars representing a wide range of food compositions. A hot-melt curtain coating technique appears commercially feasible for all foods examined; however, coatings so applied failed to withstand storage at a temperature cycling between -18 and + 40°C.

INTRODUCTION

The objective of this program has been the development of edible coating materials for dehydrated foods which provide a significant degree of protection against atmospheric moisture, oxidation and fragmentation.

This report represents the final report on Phase II of a two year program. During Phase I, a series of edible coatings was developed and tested. It was found that the effectiveness of coatings as barriers depended on the continuity of the coating as well as the conditions of storage. The continuation of this program during Phase II stressed the evaluation of commercially feasible techniques for the application of edible coatings to compressed unit servings of the following foods:

- a. dry orange juice
- b. dry non-fat milk
- c. dry cream of mushroom soup
- d. margarine
- e. dry mashed potatoes
- f. dry applesauce
- g. freeze-dried cottage cheese
- h. precooked dehydrated chicken and rice
- i. precooked dehydrated beef stew
- j. precooked dehydrated rice pudding

As in Phase I, edible coatings must be made with materials which comply with FDA regulations; coatings should yield a minimum of 3.5 Kg calories per gram; coatings should contribute no objectionable odors or flavor nor decrease the nutritional qualities of coated foods; the weight of coating material

should not exceed 16% of the weight of the coated food product.

Coatings are tested by storage at 40°C and at a cycled temperature range of -18°C to 40°C for 3 months.

Two additional studies undertaken in Phase II included the feasibility of coating foods during the terminal stage of freeze drying and the effect of absorbed gases on the sensitivity of freeze dried foods to the adverse effects of atmospheric moisture and oxygen.

SUMMARY AND CONCLUSIONS

A series of coating techniques was evaluated in an attempt to demonstrate a commercially feasible process for applying an edible, protective barrier to compressed food bars containing freeze dried foods. Of the techniques evaluated, curtain coating with a thermoplastic film appears to be commercially feasible, although existing equipment would need to be redesigned to some extent. Spraying can be used in conjunction with curtain coating in order to apply an oxygen barrier to bars previously coated with a thermoplastic moisture barrier.

Fluidized bed and dip coating techniques are not commercially feasible at the present time. Deficiencies in the properties of edible coating materials are a major problem in both of these coating techniques.

The application of thermoplastic coatings during the terminal stage of freeze drying is not feasible. A continuous coating is very difficult to obtain and impregnation of porous freeze dried foods with thermoplastic coating material prevents rehydration of the food and adversely affects the organoleptic properties of the product.

The sorption of polar and non-polar gases by freeze dried chicken did not provide any noticeable moisture barrier protection. The feasibility of this approach to protection of freeze dried foods has not been demonstrated.

Food bars that were curtain coated with an edible thermoplastic composition did not withstand the stringent storage conditions prescribed. Bars that were stored under cycling conditions ranging from -18°C to 40°C were degraded structurally within two weeks. Coated food bars exhibited greater stability at 40°C constant temperature. After two months, however, degradation of the products was evident.

The failure of coated foods stored under cycling temperature and humidity conditions is attributed to: 1) structural weakness in the coating at edges, corners and centerline of the bars - the latter being caused by the necessity of coating each side separately with the available equipment, and 2) the expansion and contraction of food bars under cycled temperature and humidity conditions, leading to breakdown in the structural integrity of the bars.

EXPERIMENTAL METHODS

Materials

A. Foods

Dehydrated foods were procured as shown in the following list:

<u>Food Item</u>	<u>Brand Name or Source</u>
1. Dry orange juice	Plant Industries, Inc
2. Non-fat dry milk	"Carnation" Instant non-fat dry milk
3. Cream of mushroom soup	"Knorr" cream of mushroom soup
4. Margarine	"Blue Bonnet"
5. Dry mashed potatoes	"Pillsbury" mashed potato flakes
6. Dry applesauce	Valley Evaporating Co
7. F.D. Cottage cheese	Freeze-dry Products, Inc
8. Precooked dehydrated chicken	Canada Freeze-dry Foods, Ltd
9. Instant Rice (for chicken rice bar)	"General Foods" Instant rice
10. Precooked dehydrated beef stew	Armour & Co
11. Dehydrated chocolate pudding (replacing rice pudding)	"Royal" Chocolate Pudding
12. Chicken soup mix (for chicken & rice bar)	"Lipton" Chicken Soup Mix
13. F.D. Halved strawberries	Canada Freeze Dry Foods, Ltd
14. F.D. 1/8" sliced carrots	Canada Freeze Dry Foods, Ltd

B. Chemicals

1. Ethyl cellulose, Std Ethoxy, Vis 50	Dow Chemical Co
2. Myvacet 5-00 (acetylglyceride monostearate)	DPI
3. Calcium stearate	American Cyanamid Co
4. Beeswax	"Beehive Brand" Will & Baumer Candle Co, Inc

5.	Glycosperse TS-20	Glyco Chemicals
6.	Glycowax S-932	
7.	Amylose laurate	(ADM Lab preparation)
8.	Amylose	A E Staley Mfg Co
9.	Gelatin 250 Bloom	Atlantic Gelatin
10.	Sodium caseinate	Land O'Lakes Dairies
11.	Fluorescein	Nutritional Biochemicals

Compression of Food Bars

Most foods were compressed with the aid of one of the following binders:

Pillsbury Binder (FD-37, final report contract DA19-129-AMC-2103, Jan. 1966)

47.1% shortening (Wecobee S Drew Chemical Co)

19.2% sodium caseinate (Land O'Lakes)

33.7% dextrin (National Starch)

100 g of ingredient mix were emulsified in 1000 cc water and the mixture was freeze dried. The dried product was ground to pass a USBS #60 sieve.

Evans Research Binder (FD-14, final report contract DA19-129-AMC-2111, August 1965)

99% lactose (Foremost Dairies)

1% CMC (7MP) (Hercules Powder Co)

100 g of ingredient mix were dissolved in 700 cc water and the mixture was freeze-dried. The dried product was ground through a USBS #60 sieve.

Binders were added at 15-20% by weight of the food product to be compressed. The ingredients for each product were blended together with binder in a Hobart

mixer. Unit servings were weighed and transferred to a 3.0" x 1.5" stainless steel compression mold. Blocks were molded with the aid of a Carver press according to conditions summarized in Table I. Formulation of food blocks was as follows:

Mashed Potatoes

100.0 g Dehydrated Potato Flakes (Pillsbury)

5.0 g Salt

0.5 g White Pepper

8.0 g Shortening (Wecobee S)

5.0 g Non-fat Dry Milk

114.0 g

Chicken and Rice

57.2 g Freeze-dried Cooked Chicken 1/4" dice

5.8 g Lipton's Chicken Soup Mix

37.0 g Minute Rice (General Foods)

100.0 g

Beef Stew

35 oz Freeze-Dried Vegetables with Gravy Mix

12 oz Freeze-Dried Precooked Beef Cuts and Snap Beans

47 oz

Preparation of Coatings

Films of various compositions were prepared by several laboratory procedures for evaluation of characteristics.

1. Casting from solvents

The ingredients of the composition were dispersed in solvent with stirring and application of heat. An aliquot of the dispersed mixture is placed at one

end of a 12" x 12" Plexiglass plate and an adjustable draw bar is used to draw a film. Solvent is removed by a current of warm air directed at the plate and the dry film is peeled from the Plexiglass plate.

2. Formation of Films From Hot Melts

Two methods were used to make films from hot melts: a) films were drawn on a chrome plate with a draw bar; b) hot melts were pressed to desired thickness between chrome plates with a heated press.

The hot melts used in this study, as in Phase I, consisted of ethyl-cellulose acetylated glycerides and plasticizers. The acetylated monoglyceride is melted first on a silicone oil bath. Ethyl cellulose and plasticizers are added to the melt and the bath temperature is increased to 300°F. The melt is mixed continuously to prevent scorching. The draw bar is heated to 275°F and an aliquot of the molten mixture is poured on a heated chrome plate. The chrome plate is set on a vacuum platen to keep the chrome plate stationary while the film is being drawn. The drawn film is allowed to cool before being peeled from the plate.

Pressed films were made on a hydraulic press equipped with steam heated platens. The platens are heated to 250°F and a small amount of hot melt is placed between chrome ferrotypic plates. The plates are pressed for 3 minutes at a pressure of 2000 psi. The pressure is released and the plates are cooled before the film is peeled off.

Better control of film thickness was obtained with the heated press than could be obtained by drawing films with a heated bar.

3. Sprayed Coatings

Protein coatings were applied to moisture-barrier films by spraying.

Aqueous dispersions of sodium caseinate were sprayed with a DeVilbis type MBC

spray gun equipped with AV-15-EX nozzle.

Sodium caseinate and glycerol plasticizer were dispersed in water by means of an Osterizer blender. The dispersion was vacuum deaerated and filtered through glass wool to remove air bubbles and undispersed material that could cause discontinuities in films.

Food bars to be spray coated were set on rubber stoppers and placed on a slowly rotating turntable. The tops and sides of bars were sprayed and dried in a forced air oven at 35°C for approximately 10 minutes. The bars were turned over and the other side was sprayed in the same manner. Bars were visually inspected for film continuity by difference in sheen between the protein coating (lustrous) and the moisture barrier (dull).

Application of Coatings to Foods by Commercial Methods

1. The Wurster Fluidized Bed Coating Process

The Wurster process was developed by the Wisconsin Alumni Research Foundation* for coating pharmaceutical tablets. The Wurster process is a batch process whose size depends on the diameter of the bed or column. Columns from 6 to 18 inches in diameter are available. Coating material is dissolved in an appropriate solvent such as chloroform, methylene or ethylene chloride, and sprayed on particles suspended in the column by an upward stream of hot air.

Freeze-dried half strawberries were coated with ethyl cellulose - amylose laurate, and with ethyl cellulose - Myoset 5-00 - calcium stearate compositions dissolved in chloroform. Between 15 and 30% coating based on the weight of strawberries was applied in 5.6 to 14.5 minutes. The size of freeze-dried strawberry batches ranged from 195 to 235 grams. A 6-inch diameter column was used in the coating process. Air inlet and outlet temperatures averaged 127°F and 100°F, respectively.

* The cooperation of Dr John Birdsall and WARF in conducting these tests is greatly appreciated.

2. Curtain Coating

A. The "Eastoflow" Process

The Eastman Chemical Company* has developed a coating machine designed to extrude a curtain of melted thermoplastic coating material. The thermoplastic curtain is passed over a stationary, perforated vacuum platen on which lay the products to be coated. The curtain is drawn around the edges of the product by means of the vacuum and forms a skin tight coating. The thickness of the curtain, and of the resulting coating, is controlled by the clearance between the jaws of the extrusion head and the speed of the traversing curtain. All parts of the machine, except the vacuum platen, are mounted on rollers which allow the curtain to move across the stationary samples. The amount of vacuum drawn through the perforated platen is controlled by a valve. The operating machine is shown in Figure 1.

Fifteen pounds of ethyl cellulose, Myvocet 5-00 and calcium stearate in a ratio of 37:48:15, respectively, was prepared as follows: Myvocet 5-00 was melted in a Hobart mixing bowl and the ethyl cellulose added gradually to obtain a putty-like mix. The blend was placed in the reservoir and the heaters set at 300°F. After melting, the pump was turned on to circulate the hot resin through the extrusion head. When the mix was melted and the curtain was free from lumps or breaks, calcium stearate was added to the resin.

* "Eastoflow" is the trade name for the machine and process developed by Eastman Chemical Corp whose cooperation in coating food bars is gratefully acknowledged.

Film thickness was tested by passing the extrusion head over a cardboard of known thickness and measuring the increase in thickness.

Food bars were placed in rows on the perforated platen approximately 2.0" apart. Moderate vacuum was applied to the platen and the machine was passed over the bars a single time. The film was allowed to cool for several minutes before each bar was cut along its edges to separate it from the platen. Excess film was peeled off of the platen and returned to the heated reservoirs. The food bars were turned over and the second side was coated. The coating was allowed to harden at room temperature before rough edges of the bars were trimmed. Sodium caseinate dispersions were applied to food bars by spraying.

B. The "Liquifilm" Coating Machine

Crompton & Knowles Corp, Packaging Machinery Division, has developed a machine for the application of hot-melt films by a combination of spraying and curtain coating. A high speed chain conveyor carrying food bars through the melted coating reportedly permits complete coating in a single pass.

A laboratory scale "Liquifilm" unit was used at Crompton & Knowles research facility in Bellwood, Illinois. Trials on this machine were unsuccessful because of difficulties in controlling film thickness and hot-melt temperature, which resulted in degradation of coating material.

3. Fusion of Powdered Coatings During The Terminal Phase of Freeze Drying

Four coatings evaluated during Phase I exhibited good moisture barrier characteristics. These coatings include:

- 1) Ethylcellulose-acetoglyceride Composition
29.4% Ethylcellulose
58.8% Myvacet 5-00
5.9% Drewpol 6-2-S
5.9% Drewpol 6-2-O

- 2) Myvacet 5-00
- 3) Sunbleached white beeswax
- 4) Confectionary hard butter (Wecobee S)

All coatings were frozen in dry ice and ground to pass a USBS #16 sieve. Lean beef round was cut into 1/2 x 3/4 x 3/4" pieces and coated with 1.0 g of powdered coating per piece. Samples were freeze-dried in a Repp Sublimator Model 14 freeze drier to less than 3% moisture. Shelf temperature was adjusted to 175-200°F during the final hour of drying. This temperature range is higher than the melting points of all coatings except the ethylcellulose-acetoglyceride composition. After the coatings had fused, the shelf temperature was lowered to normal ambient temperature before breaking the vacuum. This cooling time was required to prevent the soft coatings from being drawn into the porous meat chunks. Coated pieces were transferred to a desiccator and held for storage and analysis.

4. Coating by Dipping in Dispersions and in Hot Melts

Exploratory work on coating food bars by dipping in toluene dispersions of amylose laurate and ethyl cellulose was carried out. Food bars were dipped briefly in the fluid coating mixture at room temperature, then dried in a stream of warm air.

Dip coating with a melt of ethylcellulose-Myvacet 5-00-calcium stearate was carried out at a melt temperature of 275-300°F.

5. Coating of Margarine by Dipping in Hot Melts

Frozen margarine shaped in balls was dipped in hot melts of Myvacet 5-00, sodium caseinate, and beeswax and left at room temperature until the coating hardened. Wooden applicators were used to hold the margarine balls. After the coating hardened the applicators were removed and bare spots on margarine balls were touched with melted coating.

Physical and Chemical Tests

1. Fragmentation of Bars

The effect of coatings on fragmentation of food bars was determined by dropping bars from a height of six feet onto the floor.

2. Tensile Strength of Films

Tensile strength of unsupported films was determined according to ASTM 882-56T by means of an Instron Model TT-C Universal Testing Machine.

3. Elongation

Elongation was calculated in percent from tensile strength data at the break point.

4. Moisture Vapor Transmission

Films were tested for moisture vapor according to the procedure reported in Phase I of this study. This is a modified MVT can analysis where the film is exposed to a relative humidity differential of 90% at a constant temperature of 32°C for a period of 24 hours.

5. Oxygen Permeability

Oxygen permeability of films was determined by the method of Karel et al¹⁾. A number of test cells were connected in series to allow evaluation of up to 6 films simultaneously. Films were mounted in the cells between neoprene gaskets which prevented leakage at gas pressures up to 4 atmospheres. Oxygen and nitrogen flowing into the test cells were separated by the test film. The total pressure differential across the film was zero while the oxygen partial pressure differential was one atmosphere. Changes in pressure after gas samples were withdrawn were avoided by injecting 1 ml of nitrogen into the test compartment to replace the 1 ml sample of oxygen that was taken. Up to 10 samplings were taken from the same test cell without affecting the accuracy.

1) Karel et al, Food Technology 17, 91-94 (1963)

of the determination.

Gas analyses were carried out on a dual column, dual detector Fischer Model 25V gas Partitioner.

Operating conditions were:

Sample	1 ml
Column temp	75°F
Carrier gas-rate	Helium at 80 cc/min
Recorder	5 m.v. Sargent Strip Chart
Sensitivity	10 u l O ₂ at 7 ma
Retention Time	O ₂ 71 sec, N ₂ 97 sec

Permeability of films was determined from the formula $P = YXC/tp$ where P is permeability constant expressed in units of $\frac{CC \times mils}{m^2 \times atm \times day}$; Y is the cell constant calculated as the ratio of cell volume to film area; X is film thickness in mils; C is the volumetric % concentration of oxygen in the sample; t is time in days; and p is the partial pressure differential, in atmospheres, of oxygen across the film.

6. Equilibrium Relative Humidity

ERH was determined as described in Technical Report FD-9, Contract No DA19-129-AMC-11(N), with the following modifications: one quart jars with #12 rubber stoppers were used in place of Mason jars; polyethylene cylinders approximately 1-1/8" ID and 1" long were suspended in the jars by means of nylon thread. Samples were placed in the polyethylene cylinders and their change in weight determined as a function of time.

Constant R. H. conditions were obtained through the use of the following solutions:

H ₂ SO ₄ sp.g.	1.53	10% R H
Saturated Na ₂ CrO ₄		50
Saturated NaCl		75
Saturated CaSO ₄		98%

R H was determined by means of a Minneapolis Honeywell W611A relative humidity indicator. Tests were run at 73° and 105°F.

7. Moisture Analysis

The moisture content of coated and uncoated foods was determined by vacuum oven at 60°C and 18" vacuum. The moisture content of films and coatings was determined by the Karl Fischer titration method²⁾

2) Fischer, K, Angew Chem 48, 394-6 (1937)

8. Peroxide Value

The degree of fat oxidation was determined by the iodometric method for peroxide value described by Privett et al³⁾

9. Total Carbonyl

Carbonyl compounds developed during the storage of coated food bars were determined by the Shell Development Co method described in Emeryville Method Series EMJ 3038/64.

Traces of carbonyl compounds in extracted fat were measured spectrophotometrically by treating a sample with an alcoholic solution of 2,4-dinitrophenylhydrazine and hydrochloric acid. Formed hydrozones were converted to red-colored compounds by treating with caustic solution. The amount of carbonyl compounds present in a sample was determined from a standard curve and expressed as % C=O on a weight basis.

3) Privett et al, JAOCS, 30, 17-21 (1953)

10. Organoleptic Evaluation

Food bars were evaluated organoleptically after coating. Flavor and color were the significant characteristics that were evaluated. Recipes for evaluating products were obtained from Technical Documentary Report No AMRL-TDR-64-38, pp 21-26 (1964) (Table 2).

11. Film Thickness

A General Electric thickness gauge Type B was used to measure film and coating thickness.

12. Continuity of Coatings

The continuity of thermoplastic coatings fused onto freeze dried meats during the freeze drying cycle was determined by means of a staining technique. Cooked and uncooked cubes of meat were sprayed with a 0.1% (w/v) solution of fluorescein before depositing the powdered thermoplastic coating materials, as described above. The coated freeze-dried meat was placed on the stage of a Bausch and Lomb 3 dimensional binocular microscope and illuminated with ultra-violet light at 3660°A. Uncoated areas of the sample fluoresced while coated areas appeared dull gray in color.

Storage Conditions

1. 40°C and 78% \pm 7% RH

Coated food bars were stored in a constant temperature box maintained at 40°C (104°F). Relative humidity was maintained at 48 \pm 7% by means of 83% propylene glycol in water. Food bars were placed on a screen that had been covered with cheese cloth. Six to 9 blocks of each product were placed in storage and 2-3 blocks removed at each evaluation period.

2. Cycling Temperatures of 0°F to 104°F.

A Tenney Model T30UFR environmental Chamber equipped with a Bristol series 500 temperature programmer was used for cycling, storage conditions. Cams were cut to provide 2-24 hour periods at 0°F separated by two periods at 104°F for 24 hours and 64 hours respectively. Eight hours were required to obtain equilibrium at each new condition. Relative humidity was 50-60% at 104°F and increased to 85% as the temperature fell towards the freezing point. Below 0°C, the relative humidity approached 100%.

Treatment of Freeze Dried Meat with Polar Gases

Weighed samples of freeze-dried diced chicken were dried under vacuum to constant weight. The drying chamber was sealed off from the vacuum pump and hydrogen chloride gas (Matheson) was bled in to obtain a partial pressure of 200 mm. As the HCl gas was absorbed, more gas was admitted to maintain the 200 mm partial pressure. Absorption was complete within one hour. The vacuum was then released and the flask and sample reweighed to determine the amount of absorbed gas. It was assumed that when the partial pressure of the gas in the flask remained constant, an equilibrium was reached between the free gas in the flask and the gas that had been absorbed by the sample.

Gassed samples and controls were exposed to atmospheric conditions (77°, 50% RH) and their rates of moisture uptake were determined.

The above procedure was also used with Freon C-318 (perfluorocyclobutone). Equilibration with Freon C-318 required only 5 minutes compared to one hour for HCl gas.

RESULTS AND DISCUSSION

Properties of Moisture Barrier Films.

It was concluded from Phase I that hot melt compositions based on ethyl-cellulose and acetylated monoglycerides performed well as moisture barriers and as protection against fragmentation. The composition developed in Phase I contained polyglycerol esters as plasticizers. Polyglycerol esters proved to be unstable at temperatures required to form and to apply the hot melt and they were replaced with calcium stearate which functions as a plasticizer, reduces tackiness and increases thermal stability of the hot melt.

Various ratios of calcium stearate, ethylcellulose and acetylated mono-glycerides were evaluated for compatibility and performance (Table 3). Films composed of ethylcellulose and amylose laurate were also evaluated (Table 3). Solvent-cast films composed of ethylcellulose, acetylated monoglyceride and calcium stearate did not change appreciably in tensile strength, elongation or moisture transmission as the ratios of the films' components were changed.

A hot pressed film containing 37% ethylcellulose, 48% Myvocet 5-00 and 15% calcium stearate exhibited the lowest moisture vapor transmission and this composition was used in coating food bars. The lower moisture vapor transmission obtained with the latter film is attributed mainly to the greater film thickness.

Films composed of ethylcellulose plus amylose laurate were also cast from solvent. Efforts to prepare these films from hot melts were not successful because of the poor heat conductivity and lack of plasticity of this coating composition. The moisture vapor transmission of solvent-cast films did not change when the ratio of amylose laurate to ethylcellulose was increased (Table 3). These films exhibit much higher tensile strength and elongations than do the ethylcellulose, acetylated monoglyceride compositions.

Properties of Oxygen Barrier Films

Studies carried out in Phase I indicated that sodium soy proteinate films were effective oxygen barriers. Sodium caseinate films are also effective and are more easily prepared. In the present study sodium caseinate was dispersed in water with agitation and bubbles were removed by vacuum de-aeration.

Sodium caseinate films were plasticized with glycerol. Films were prepared from dispersions containing 15% sodium caseinate and 1.25 to 5% glycerol, based on the weight of water. The characteristics of these films are given in Table 4. Splitting was frequently encountered in films which contained the lower level of plasticizer (1.25%). Films prepared with 5% glycerol and 1.9 mils in thickness were completely permeable to oxygen after reaching equilibrium in 250 hours (Fig 2). Decreasing the glycerol level to 2.5% significantly reduced the permeability of sodium caseinate film to oxygen (Fig 2, Table 4).

The addition of gelatin to dispersions of sodium caseinate did not result in improvement in oxygen barrier characteristic over films prepared with sodium caseinate alone (Table 4).

It is evident that glycerol retards moisture evaporation from the film, thereby maintaining film continuity. The oxygen and nitrogen gases used in the oxygen permeability test were not humidified before being passed into the test cells. The continuous flow of dry gas across the film surface probably had a drying effect which accelerated film failure by cracking.

Equilibrium relative humidity data on sodium caseinate films is given in Table 5 and Figure 3. Sodium caseinate film containing 7.57% moisture and 4.4% glycerol reached equilibrium after 22 hours at 50% and 75% R.H., 23°C. Film equilibrated at 98% R.H. developed mold growth after 94 hours. No cracking

was observed in sodium caseinate films equilibrated at 50% or 75% R.H.

Based on the above data, aqueous dispersions containing 15% sodium caseinate and 2.5% glycerol were used to coat food bars that had been previously coated with a moisture barrier.

The oxygen permeability of the moisture barrier film applied by curtain coating is shown in Figure 4 and Table 4. The ethylcellulose, acetylated monoglyceride, calcium stearate composition itself cannot be considered an effective oxygen barrier. The latter composition is somewhat more effective than mixtures of ethylcellulose plus amylose laurate, and ethylcellulose plus calcium stearate, whose permeabilities are shown in Figure 5.

Application of Coatings to Freeze-Dried and Compacted Foods

1. Fluidized Bed Coating

Trials with the Wurster fluidized bed coating process pointed out several disadvantages of this coating technique:

- a. It is most applicable to pieces having curved, smooth surfaces, such as peas and beans, that do not stack and are easily separated.
- b. Fragile freeze dried foods are readily fragmented by attrition in the fluidized bed. Strawberry seeds were abraded off of freeze-dried strawberry halves and in turn increased the degree of attrition.
- c. Coatings are applied in a solvent in the Wurster process. The porosity of most freeze-dried foods enables the coating to be drawn to the interior of food particles, increasing the ratio of coating to food and interfering with rehydration of foods.
- d. Solvent which penetrates into porous freeze-dried foods is difficult to remove. These solvents can cause redistribution of lipids in freeze-dried meats, thereby increasing their susceptibility to oxidation.
- e. The coating thickness is difficult to control on rough, porous surfaces of freeze-dried foods.

In spite of the above objections, continued research on the fluidized bed coating technique is warranted if coated discreet food particles are desired. Future research in this area should seek to eliminate solvent-dispersed coatings. Low melting thermoplastic materials may be applicable to the fluidized bed coating technique.

2. Curtain Coating

Curtain coating with edible thermoplastic film forming compositions appears to be commercially feasible, although considerable additional effort would be

required to adapt existing equipment to this application. Curtain coating is more readily adaptable to food bars, such as those used in the present study, than to small pieces of freeze-dried food.

Control of film thickness was not precise in the "Eastoflow" process because of variation in the hot melt flow. Film thickness ranged from 5.0 to 15.0 mils. Thickness was controlled by the width of the slit in the extrusion head and the curtain speed.

Improved thermoplastic compositions are needed to extend the pot life of the coating material. The stability of the recirculating hot melt can be extended by coating in an inert atmosphere. The Eastoflow machine would require modification to allow operation in a nitrogen atmosphere. A picture of the Eastoflow machine is shown in Figure 1. Modification of the Eastoflow machine would also be needed to allow coating of both sides of a food bar at a single pass.

Factors observed to be important in coating food bars by the Eastoflow process include the height of the food bar (distance from the top surface of the bar to the vacuum platen), the distance between the bars, and the level of vacuum drawn through the platen. Thicker food bars were more difficult to coat than were thinner bars. Sufficient distance between bars is required to permit the vacuum to pull the curtain tightly around the bars. The need for sufficient vacuum to allow molding of the film to the bar must be balanced against excessive stretching of the film, particularly at the edges and corners of food bars.

The presence of air bubbles in the hot melt interferes with the continuity and smoothness of the coating. The Eastoflow machine is designed to prevent the occurrence of air bubbles in the film.

The hot melt temperature was controlled at 300°F for best flowability with the ethylcellulose-acetylated monoglyceride-calcium stearate coating composition. This high temperature resulted in degradation of calcium stearate over a period of 3 hours. The film color, initially light tan, gradually turned brown.

Continuous coatings were obtained on most compressed food bars. High protein bars including non-fat dry milk and freeze-dried cottage cheese were more difficult to coat. Splitting was encountered at the edges of these bars after they had been sprayed with sodium caseinate dispersions. Apparently, the thermoplastic coatings do not adhere well to highly polar protein foods and a good bond between coating and food surface was not obtained.

3. Dip Coating

Coating freeze-dried foods by dipping in hot melts or solvent-thinned coatings has serious disadvantages. Only exploratory experiments were carried out to establish the feasibility of these techniques. Dried food bars that were dip-coated in a solvent thinned dispersion of ethylcellulose, acetylated monoglyceride and calcium stearate required 24 hours in a warm air stream to reduce the solvent to a level that could not be detected olfactorily. Coating food bars by dipping in hot melts was not successful because of the high viscosity of the coating material. Film thickness and uniformity could not be controlled. Dip coating is analogous to enrobing. Enrobing requires coatings with low melting points. This type of coating operation is feasible with glycerides but not with cellulosic materials which exhibit high softening points and high viscosity.

4. Coating of Margarine

The coating of margarine was considered separately from the coating of food bars because of the special properties of margarine. A margarine coating must have sufficient structural strength to withstand the internal pressure that develops when the melting point of margarine is exceeded. Oxygen barrier protection is also required.

Spray application of sodium caseinate film on margarine was not feasible because of the poor spreadability of aqueous dispersions on fat. Use of a surfactant (polyoxyethylene 20 monostearate, Tween 60) at a level of 0.1% in a sodium caseinate dispersion did not improve its spreadability or adhesion to margarine.

Coatings prepared from mixtures of acetylated monoglyceride, sodium caseinate, and beeswax on hard coating butter were prepared as follows:

<u>Ingredient</u>	<u>Weight</u>	<u>Preparation</u>	<u>Properties</u>
Myvocet 5-00 Sodium caseinate Beeswax	275 g 180	Melt Myvocet, blend in caseinate, add beeswax	Smooth paste solidifies at room temp
Myvocet 5-00 Sodium caseinate Beeswax	250 125 75	and continue heating and mixing to form a paste	Fast setting at room temp. Hard coating
Myvocet 5-00 Sodium caseinate Glycerol	325 200	Melt Myvocet, suspend some caseinate in glycerol and remainder in Myvocet, mix at high speed	Slower in hardening than other coatings
Myvocet 5-00 Sodium caseinate Glycowax S-932	200 125 50	Melt Myvocet, disperse caseinate, add Glycowax	Slow in hardening

Coatings prepared from the above mixtures were applied to frozen slices or balls of margarine. Margarine balls and slices averaged 25 g. Frozen pieces of margarine were held on applicator sticks and dipped into the hot

melt coatings. Margarine balls coated with the composition containing 325 parts Myvocet 5-00, 200 parts sodium caseinate and 20 parts glycerol withstood 24 hours of storage at 37°C, but cracked within 6 hours at 40°C. Samples stored at 30° did not show any leakage over a period of two weeks.

Cracking of coated margarine slices occurred at edges and corners where the coating was thinner. In margarine balls, cracking occurred on the bottom surface where flattening occurred following storage at elevated temperatures. It is believed that the internal pressure of coated balls and slices of margarine increases when the margarine melts and releases gas that had been entrapped in the emulsion. The mechanical strength of the coating is probably further weakened by migration of fat into the coating.

Pharmaceutical grade gelatin capsules were evaluated as a means of packaging margarine. Size #10 capsules averaged 20 cc in volume and 4.1 g in weight. Capsules that were filled with margarine and sealed with melted beeswax leaked when stored at 40°C.

Quarter pound bars of margarine were coated with ethylcellulose, acetylated monoglyceride and calcium stearate hot-melt mixture with the aid of the Eastoflow curtain coating machine. The bars were not frozen prior to coating and some oiling-off occurred. Of two bars coated by this technique, one remained intact at 40°C for one week, but exhibited cracking after 8 days at this temperature. The appearance of these samples was undesirable because of oil separation after storage at 40°C. No off-odors were detected in the product after storage.

It was suggested that a preformed sleeve of the ethylcellulose composition might allow packaging of margarine bars while avoiding the effect of high coating temperatures on the margarine. Attempts to form a continuous sleeve of the ethylcellulose composition in an aluminum mold were not successful because of the poor flowability of this composition.

Coating During The Terminal Stage of Freeze Drying

Pieces of beef and chicken were coated with powdered thermoplastic materials and freeze-dried as described. The fusion of powdered lipid materials on meat surfaces did not result in formation of continuous coatings. The problems encountered with this approach were primarily mechanical. Sides and bottoms of food pieces lying on trays were difficult to coat. By placing an excess of thermoplastic on the top surface of food pieces, some would melt down the sides. High ratios of coating to food caused difficulty in rehydrating the partially coated food. The irregular surface of freeze-dried meat emphasized the problem of coating filling the crevices and finding its way into the interior portions of freeze-dried meat samples.

Freeze-dried meat that had been coated with Wecobee S hard coating butter, beeswax and Myvacet 5-00 acetylated monoglyceride were stored at 50%, 75% and 98% relative humidities at 23°C. The rates of moisture uptake under these conditions are shown in Figures 6 to 8. Since the ratio of coating to food could not be closely controlled, results are reported in these figures on an as is basis. Ether extraction of coated samples indicated the following:

<u>Coating</u>	<u>% Ether Extractibles</u>
None	7.1
Myvacet 5-00	37.7
Wecobee S	58.8
Beeswax	43.6

When the moisture uptake for coated freeze-dried meats shown in Figures 6 to 8 are corrected for the weight of the coating, there is little difference between uncoated controls and coated samples.

The failure of this coating technique can be attributed to absence of a continuous coating. Samples of meat that were stained with fluorescein dye prior to freeze-drying and coating were observed to have discontinuous coatings when viewed with a stereo microscope under ultraviolet illumination.

The Effect of Gases on Moisture Sorption by Freeze Dried Chicken

The work of Benson and coworkers¹ demonstrated that both polar and non-polar gases form a monolayer on protein surfaces. Polar gases were also strongly bound to internal polar sites. The formation of chemical bonds was demonstrated in some cases.

An objective of the present study was to determine if treatment of dehydrated foods with polar gases would provide protection against adverse environmental conditions. Benson's work further showed that steric factors were of significance in determining how much polar gas could be sorbed by protein. Larger polar gas molecules did not penetrate the protein matrix easily. Thus, methylamine is not sorbed as strongly by protein as is ammonia although it is a stronger base than ammonia. Sorption of non-polar gases by protein is essentially reversible. Even with "irreversible" sorption, most of the polar gas could be removed by heating.

The requirement of edibility severely limited the choice of gases in the present study. Gases chosen for study include hydrogen chloride, Freon 21 (dichlorofluoromethane) and Freon C-318 (perfluorocyclobutane). Freon 21 is the most polar of the Freon chemicals now available. Freon C-318 is a compact

¹ R Srinivason and S W Benson, J Am Chem Soc 77, 6371 (1955); 78, 2405 (1956); 78, 5262 (1956).

S W Benson and R L Richardson ibid 77, 2585 (1955).

S W Benson and D A Ellis ibid 70, 3563 (1948); 72, 2095 (1950).

J M Seehof and S W Benson ibid 73, 5053 (1951); 75, 3925 (1953); 77, 2579 (1955).

S W Benson et al, ibid 75, 6040 (1953); 72, 2102 (1950).

J M Seehof et al, ibid 75, 2427 (1953).

cyclic molecule that is completely fluorinated and non-polar. It was felt that Freon C-318 might penetrate the structure of the protein more easily than non-cyclic Freons.

Benson showed that oxygen covered only the surface of a protein, but water vapor was sorbed in the interior of protein molecules as well as on the surface.

The effect of the polar and non-polar gases on sorption of atmospheric moisture by freeze-dried chicken pieces was studied to evaluate the practicality of gaseous barriers.

A flask containing freeze-dried chicken pieces was evacuated by means of a diffusion pump to strip off as much sorbed gas as possible. The chamber was then brought to a partial pressure of 200 mm with the gas under study. This pressure represents an estimate of the partial pressure necessary to obtain rapid saturation of most of the sorption sites. The amount of gas sorbed by the freeze-dried chicken pieces is estimated in Table 6.

Gas treated and control samples of freeze-dried chicken were exposed to 50% R.H. at 77°F. The moisture content of both control and gassed samples increased nearly 50% in 0.5 hours (Table 7, Figure 9). Moisture contents of 9-11% were obtained in the freeze-dried chicken after 24 hours at 50% R.H., 77°F.

The available data does not show a significant protective effect of the gases on moisture sorption by freeze-dried chicken pieces.

Storage Studies on Coated Food Bars

A. Storage Conditions: 40°C. 48.0 ± 7% R.H.

Although care was taken to prevent moisture uptake by food bars prior to coating, Table 8 indicates appreciable moisture gain had occurred prior to coating. Moisture gain was difficult to prevent since the coating was done in Kingsport, Tennessee during the late spring and early summer months when relative humidities were quite high. Freeze-dried cottage cheese, chicken, orange juice and applesauce were particularly sensitive.

Uncoated products with initial moistures of approximately 4.0% lost weight after 20 days in storage at 40°C, 48.0% RH, while samples with initial moisture levels below 2.0% increased in moisture from 2 to 4 times.

Most of the coated food bars lost weight after 20 days storage (Table 8). Only applesauce and chicken and rice bars increased in weight after 20 days, but the latter product lost weight after 2 months in storage.

The data shown in Table 8 indicates that the coating provided some moisture barrier protection in a number of the food products. Comparing moisture gains of coated and uncoated products, and allowing for the difference in initial moisture content, applesauce, chicken and rice, chocolate pudding and cottage cheese show lower moisture gains among coated than in uncoated bars after 20 days storage.

The protective effect of the ethylcellulose acetylated monoglyceride - calcium stearate coating is also seen in a comparison of moisture contents of the food bars after two months storage (Table 8). Cottage cheese and non-fat dry milk and chocolate pudding showed moisture increases, but the final moisture contents of 5.22, 4.55 and 1.86, respectively, are not excessive in view of the initial moisture levels of the bars. Both chicken and rice and beef stew

bars lost moisture after two months storage at 40°C, 48% R.H. Mashed potatoes and cream of mushroom soup bars also decreased in moisture content. Moisture changes in coated food bars may have been contributed to by small slits or cracks in the coating, such as was encountered in coated cottage cheese and non-fat dry milk bars. Moisture may also have been lost with the coating, which was stripped off prior to analysis.

Oxidative Deterioration

Peroxide values and total carbonyl compounds in stored uncoated and coated food bars are given in Table 9. The uncoated samples were stored for 20 days while the coated samples were evaluated after 20 and 60 days of storage. No peroxide or carbonyl compounds were detected initially in coated and uncoated food bars. While the data are too limited to be of much significance, coated food bars tend to exhibit higher indices of oxidative deterioration than do uncoated bars. One explanation for this trend is that the coating may have trapped volatile oxidation products in the bar while these products might escape more easily from uncoated bars. Another cause for higher levels of oxidation among coated bars may be the high temperature of the melted coating having accelerated the initiation of lipid oxidation. Fast cooling of coated food bars might improve their oxidative stability.

Organoleptic Evaluation

Food bars prepared with the Pillsbury-type binder described above evidenced fat separation under the coating. Separation was most pronounced in chicken and rice and cream of mushroom soup bars. Compression of beef stew bars resulted in fat pooling in pockets; there was no objectionable oiliness on the product surface, however.

All coated samples exhibited varying degrees of browning after 2 months storage at 40°C, 48% R.H. After 20 days storage under these conditions, uncoated samples exhibited a lower degree of browning than did coated samples. Applesauce and orange juice browned so badly that the reconstituted products were unacceptable. The normal flavors of the latter products were barely recognizable after 2 months storage. Cottage cheese and non-fat dry milk changed in color to dark cream or light tan and developed an off-flavor associated with stored casein. Beef stew did not show marked changes in color, but the flavor of rehydrated beef was badly oxidized. The flavor of the vegetables in beef stew did not change appreciably compared to an uncoated sample stored at room temperature.

Chicken and rice bars were fairly acceptable in color and flavor after 2 months storage at 40°C. As in the case of beef stew bars, some fat pooling under the coating was evident.

B. Cycled Storage Conditions

Food bars coated with ethylcellulose-acetoglyceride-calcium stearate composition were stored under the cycled conditions of temperature and humidity previously described. Table 10 shows that most of the products gained high levels of moisture. Within a week, the coating on all food bars had split or peeled off, exposing the bars to the conditions in the environmental chamber (Figure 10). Food bars expanded as the exposed portions absorbed moisture. In some cases, exposed food bars completely lost their structural integrity.

The surfaces of coated bars blistered after first being placed in the cycled storage conditions. Blistering is ascribed to differences in the rates of moisture sorption by sodium caseinate and the ethylcellulose composition

and the resultant greater elasticity of the sodium caseinate layer. Batches of ethylcellulose composition film with and without the sodium caseinate laminate were stored under the cycled conditions for one week. The film with the sodium caseinate laminate had blistered and the edges rolled up. The ethylcellulose composition alone remained smooth and flat under these conditions. The appearance of intact coated food bars is shown in Figure 11.

Table 1
Compression of Food Blocks

Food Item	Block Wt.	Pressure PSI	Dwell Sec	Holding Wt. & Time in Min.	Block Condition
Chocolate ² Pudding	1.0 oz	2000	15	10	good
Mushroom ¹ Soup	1.0 oz	1500	15	10	fairly good
Applesauce	37.2 gms	800	20	--	very good
Orange juice ²	1.0 oz	1100	30	10	" "
NF dry milk ²	22.7 gms	1000	30	10	good
Mashed potatoes ¹	1.0 oz	2000	15	10	good
Chicken & rice ¹	30.0 gms	1000	30	15	good
Beef stew ¹	1.0 oz	1500	30	15	fairly good
Cottage cheese ²	1.0 oz	1000	30	--	good

¹ Pillsbury binder added at 20%

² Evans Research binder added at 15%

Table 2

Reconstitution of Compressed Food Bars

<u>Product</u>	<u>Wt.</u>	<u>Water Addition</u>	<u>Recipe</u>
Mashed Potatoes	28.3 gms	90 cc	Add <u>water hot</u> . Stir until smooth and evenly mixed.
Applesauce	37.2	180 cc	Add <u>cold water</u> . Stir. Let stand 10 min. Record time for complete rehydration.
Orange juice	28.3	118 cc	Add <u>cold water</u> . Stir until mixed.
NF dry milk	22.7	236 cc	Add <u>cold water</u> . Stir until dissolved.
Cottage cheese	28.3	90 cc	Add <u>cold water</u> . Stir and mix to smooth consistency.
Beef & Vegetables	28.3	135 cc	Add <u>water hot</u> . Stir, let stand for 30 min. Evaluate. If need more time to rehydrate, add more hot water and record time.
Chicken & rice	32.0	120 cc	Add <u>water hot</u> . Follow same as in Beef & Vegetables.
Chocolate Pudding	28.3	130 cc	Add <u>cold water</u> . Stir well and cook to thickness.
Cream of mushroom soup	28.3	180 cc	Add <u>hot water</u> . Stir. Let stand for 20-25 min. Cook to thickness if needed.

Table 3

Characteristics of Moisture Barrier Films

Components	Ratio %	Thickness mil	Tensile lb/ Strength sq in	Elongation %	MVT ¹ g/sq in/24 hrs
Ethylcellulose-acetylated mono-glyceride-calcium stearate	28.57 42.86 28.57 42.86 42.86 14.28	1.0	320-420 640-655	2.0-4.0 3.5-4.5	0.492 0.508
(Hot Melt)	37.0 48.0 15.0	4.0	370-430	17.0-19.0	0.149
Ethylcellulose-calcium stearate	75.0 25.0	1.5	4050-4480	3.2-3.5	0.676
	50.0 50.0	0.6	1410-1525	1.2-1.3	0.467
Ethylcellulose-amylose laurate	75.0 25.0	1.0	5160-5220	19.5-24.5	0.539
	38.5 61.5	1.0	3320-3680	6.0-12.5	0.537

Table 4

Characteristics of Oxygen Barrier Films

Film Components	Ratio of Comp %	Film Thickness mil	% O ₂ in Test Cell			Permeability Constant
			50 hrs	100 hrs	Final	
Sodium caseinate-glycerol-water	15					
	5.0	1.7	31%	59.5%	96.0%	
	100				(334 hrs)	
	15					
	2.5	3.5	1.0%	1.0%	5.6%	233
	100				(250 hrs)	
	15					
	1.25	4.0	2.5%	4.5%	6.2%	
	100				(144 hrs)	Film split before equilibrating
Sodium caseinate-gelation-glycerol-water	14.0					
	1.0					
	1.25	1.5				
	100					
Ethylcellulose-acetylated mono-glyceride-calcium stearate	42.86					
	42.86	1.0	77.0%	81.0%	99.0%	
	14.28				(388 hrs)	
(Hot Melt)	37.0					
	48.0	4.0	21.0%	30.4%	67.7%	
	15.0				(367 hrs)	20,200
Ethylcellulose-calcium stearate	75					
	25	1.0	87.0%	88.5%	98.5%	
Ethylcellulose amylose laurate	75					
	25	2.0	78.5%	86.5%	99.0%	
					(388 hrs)	

Table 5
Moisture Uptake by Sodium Caseinate Film¹

<u>Time, hrs</u>	<u>23°C-50% RH</u>	<u>23°C-75% RH</u>	<u>23°C-98% RH</u>
0	0.6916 gm	0.6197 gm	0.7659 gm
5.5	0.7088 gm	0.6760 gm	0.9250 gm
22.0	0.7135 gm	0.8210 gm	1.3533 gm
94.0	0.7134 gm	0.8198 gm	(1.4606) ²

¹ Film contained 7.57% H₂O and 4.4% glycerol.

² Mold growth.

Table 6
Sorption of Gases by Cooked, Freeze-Dried Chicken Chunks

<u>Gas sorbed</u>	<u>g gas sorbed g meat</u>	<u>meq gas sorbed g meat</u>
Hydrogen chloride	0.0405	1.11
Dichlorofluoromethane (Freon 21)	0.0655	0.636
Perfluorocyclobutane (Freon C-318)	0.0929	0.436

Table 7

Sorption of Water Vapor by Cooked, Freeze-Dried Chicken Chunks
Treated With Polar and Non-Polar Gases

% Moisture in Chicken Meat Treated with:

<u>Time (hours)</u>	<u>Control</u>	<u>Freon 21</u>	<u>Freon C-318</u>	<u>Hydrogen Chloride</u>
0	7.79	4.62	3.36	3.76
0.5	4.86	6.74	5.51	6.47
2	6.25	6.20	7.69	6.62
4	8.60	-	7.60	5.60
6	9.23	7.97	9.34	9.04
24	9.64	10.65	9.73	8.82
48	9.96	10.45	11.88	9.35
72	-	8.55	-	-

Table 8

Storage Studies on Food Bars at 40°C, 48% RH

<u>Food Product</u>	<u>Uncoated</u>		<u>Coated</u>		<u>Final %¹</u>
	<u>Initial %</u>	<u>20 day storage %</u>	<u>Initial %</u>	<u>20 day storage %</u>	
Orange juice	1.85	3.76	4.78	3.85	2.40
Applesauce	1.86	9.13	4.51	8.57	8.16
Cottage cheese	0.89	2.96	3.51	2.17	5.22
Mashed potatoes	4.08	3.36	5.33	4.20	4.84
Chicken & rice	1.95	4.71	5.05	6.53	4.55
Beef stew	4.16	3.04	4.95	3.23	3.04
Chocolate Pudding	1.16	6.29	1.32	0.94	1.86
Mushroom soup	1.17	3.19	4.47		4.12
NF Dry milk	3.96	2.34	4.13		4.55

¹ After 2 months in storage

Table 9

Oxidative Stability of Food Bars Stored at 40°C, 48% RH

<u>Food Product</u>	<u>Uncoated¹</u>		<u>Coated</u>		<u>Carbonyl Compds %</u> <u>20 days</u> <u>60 days</u>
	<u>Peroxide Value meq/kg</u>	<u>Carbonyl Compds %</u>	<u>Peroxide Value</u> <u>20 days</u> <u>60 days</u>		
Cottage cheese	1.0	0.01	0.0	0.0	0.02 0.05
Mashed potatoes	0.0	0.01	0.0	-	0.0 -
Chicken & rice	0.0	0.02	-	3.61	- 0.06
Beef stew	3.9	0.02	11.0	-	0.04 -
Chocolate pudding	0.0	0.0	0.0	-	0.0 -
Cream of mushroom soup	0.0	0.01	-	0.0	- 0.05

¹ Data on food bars stored for 20 days.

Table 10

Moisture, Peroxide, and Carbonyl Values in Coated Food Bars

Stored at Cycling Temperatures of -18°C to 40°C for

30 Days

	<u>H₂O, %</u>		<u>Peroxide meg/kg</u>		<u>Carbonyl %</u>	
	<u>Initial</u>	<u>Final</u>	<u>Initial</u>	<u>Final</u>	<u>Initial</u>	<u>Final</u>
Orange juice	4.78	11.23	--	--	--	--
Applesauce	4.51	--	--	--	--	--
Cottage cheese	3.51	13.12	0	0.80	0	0.03
Mashed potatoes	5.33	--	0	--	0	--
Chicken & rice	5.05	24.09	0	0	0	0.05
Beef stew	4.95	19.07	0	2.85	0	0.02
NF Dry milk	--	7.51	--	--	--	--

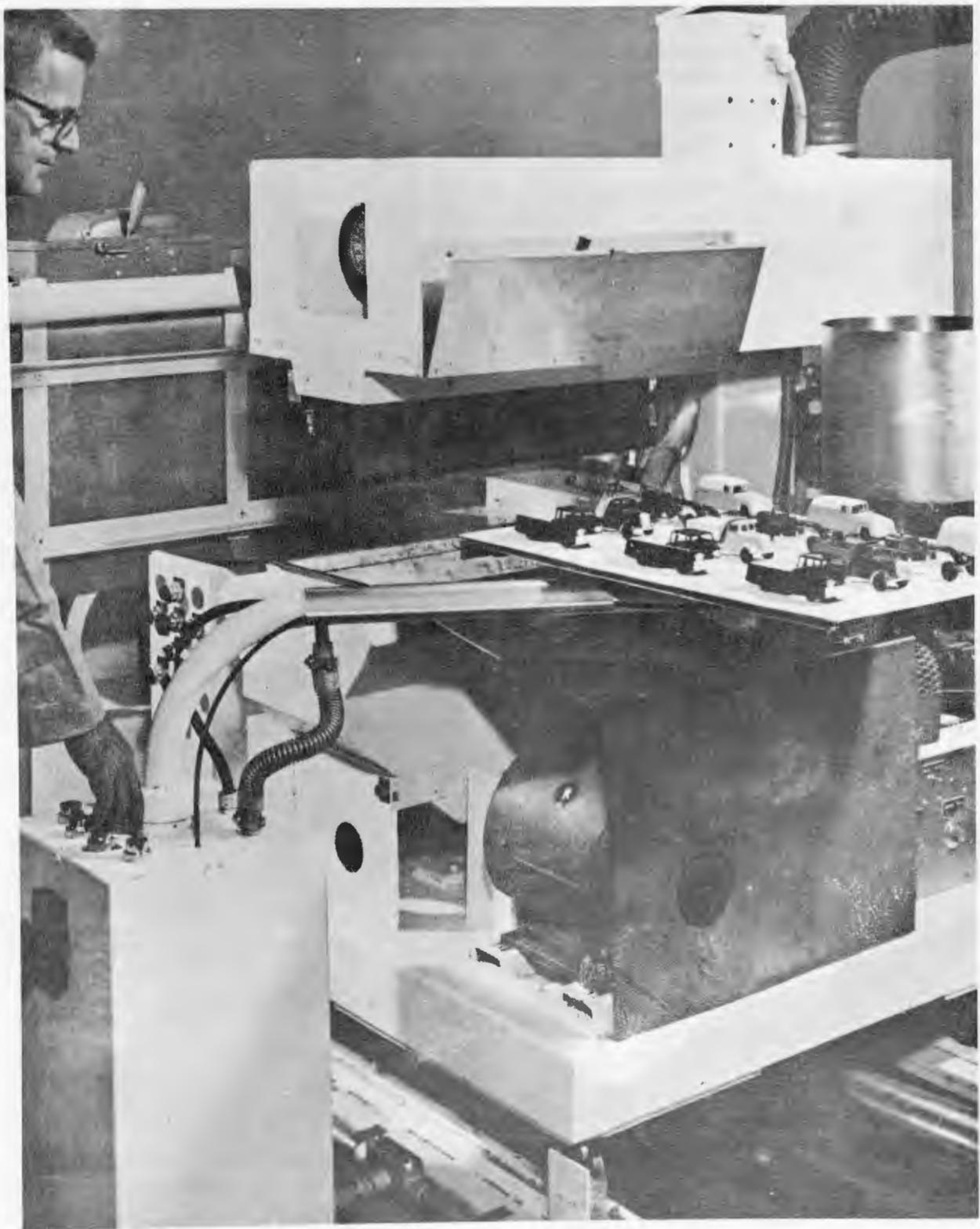


Figure 1
THE EASTOFLOW MACHINE

FIGURE 2
OXYGEN PERMEABILITY OF SODIUM CASEINATE FILMS

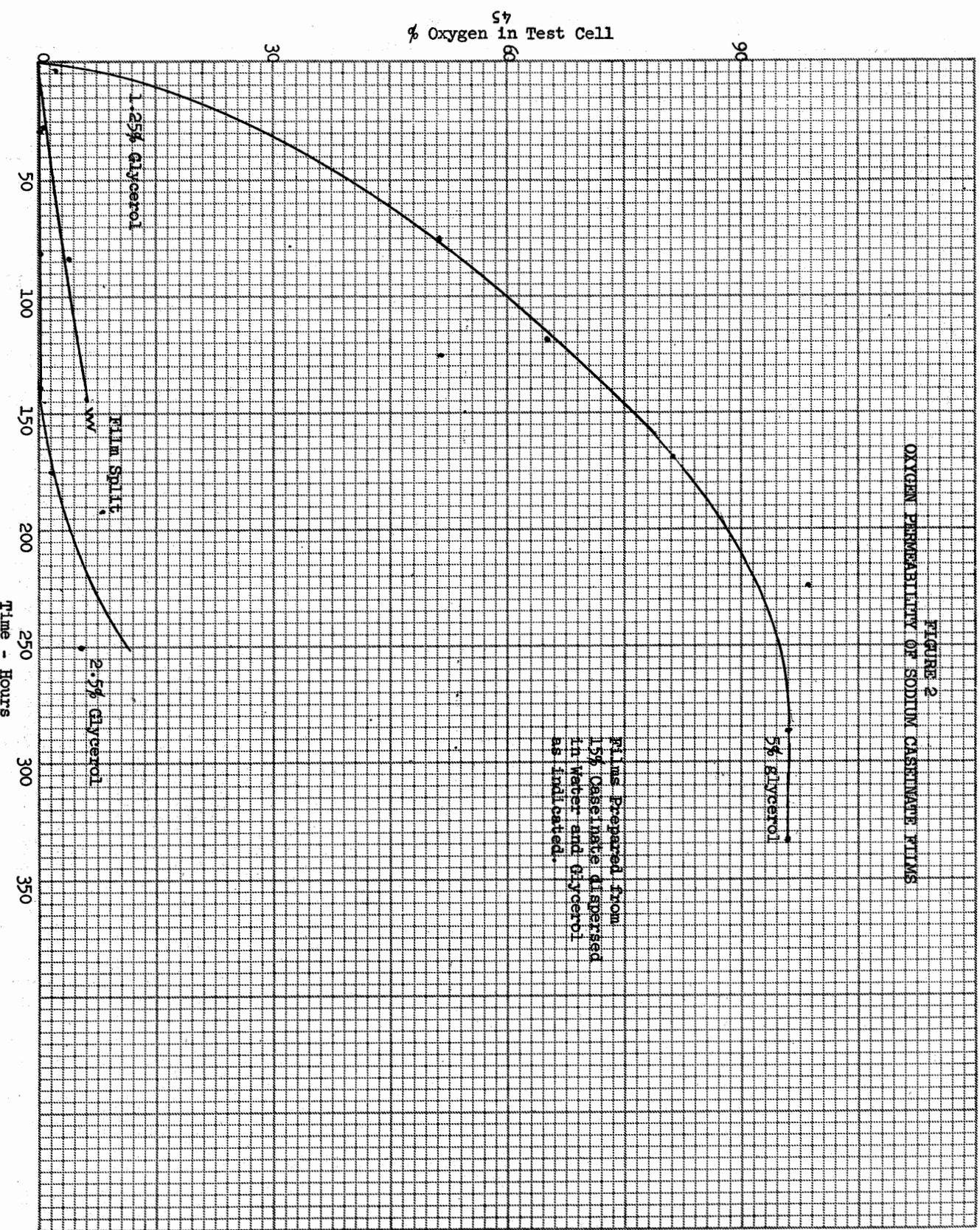


FIGURE 3
EQUILIBRIUM RELATIVE HUMIDITY OF SODIUM CASEINATE FILM

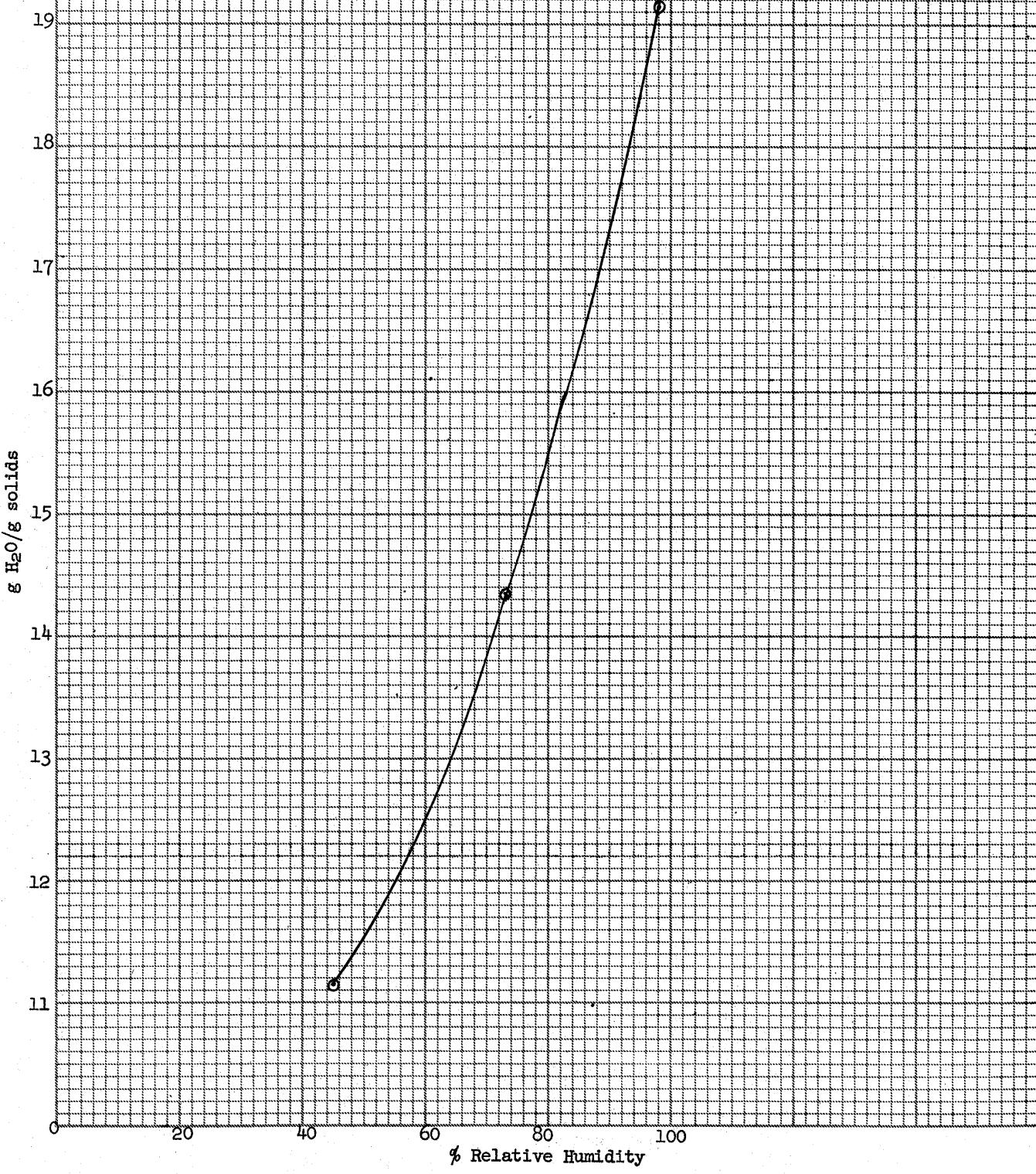


FIGURE 4
OXYGEN PERMEABILITY OF ETHYLCELLULOSE-ACETOLIC ACID-CALCIUM STEARATE FILM

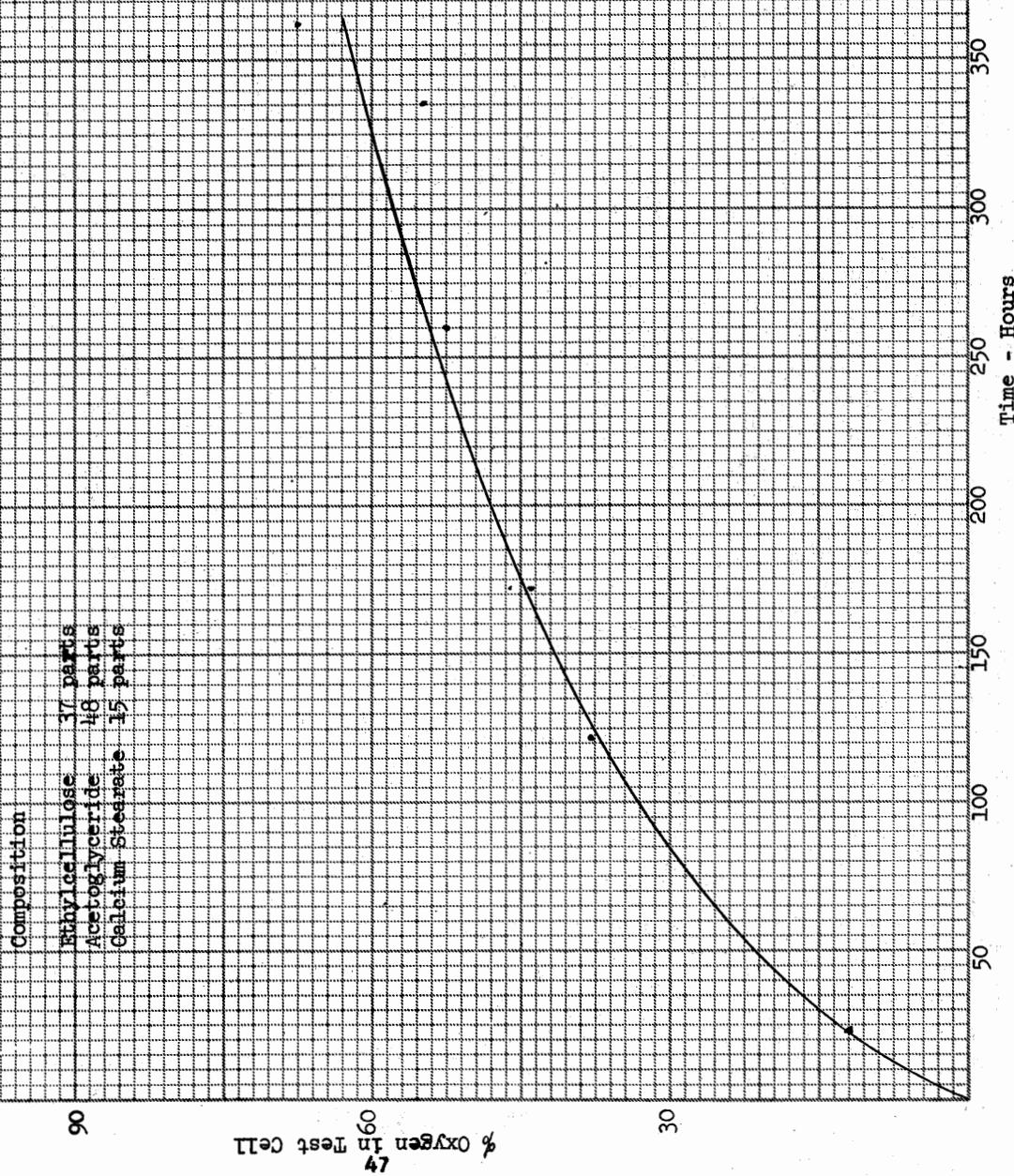


FIGURE 5
OXYGEN PERMEABILITY OF ETHYLCELLULOSE-AMYLOSE LAURATE AND ETHYLCELLULOSE-CALCIUM SULFATE FILMS

GR-4

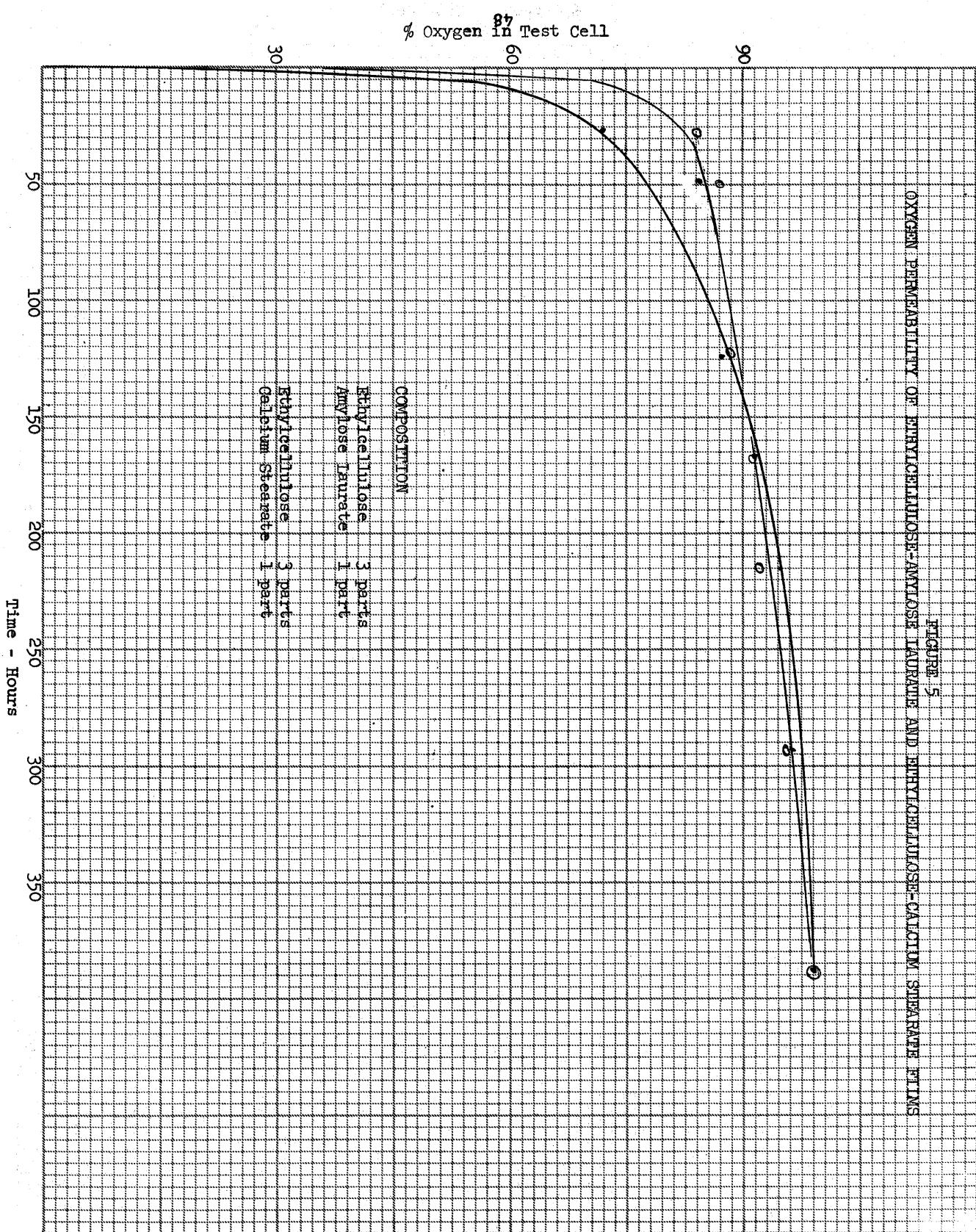


FIGURE 6
MOISTURE GAIN BY FREEZE DRIED BEEF COATED IN THE TERMINAL STAGE
OF FREEZE DRYING AND STORED AT 50% RH, 23°C.

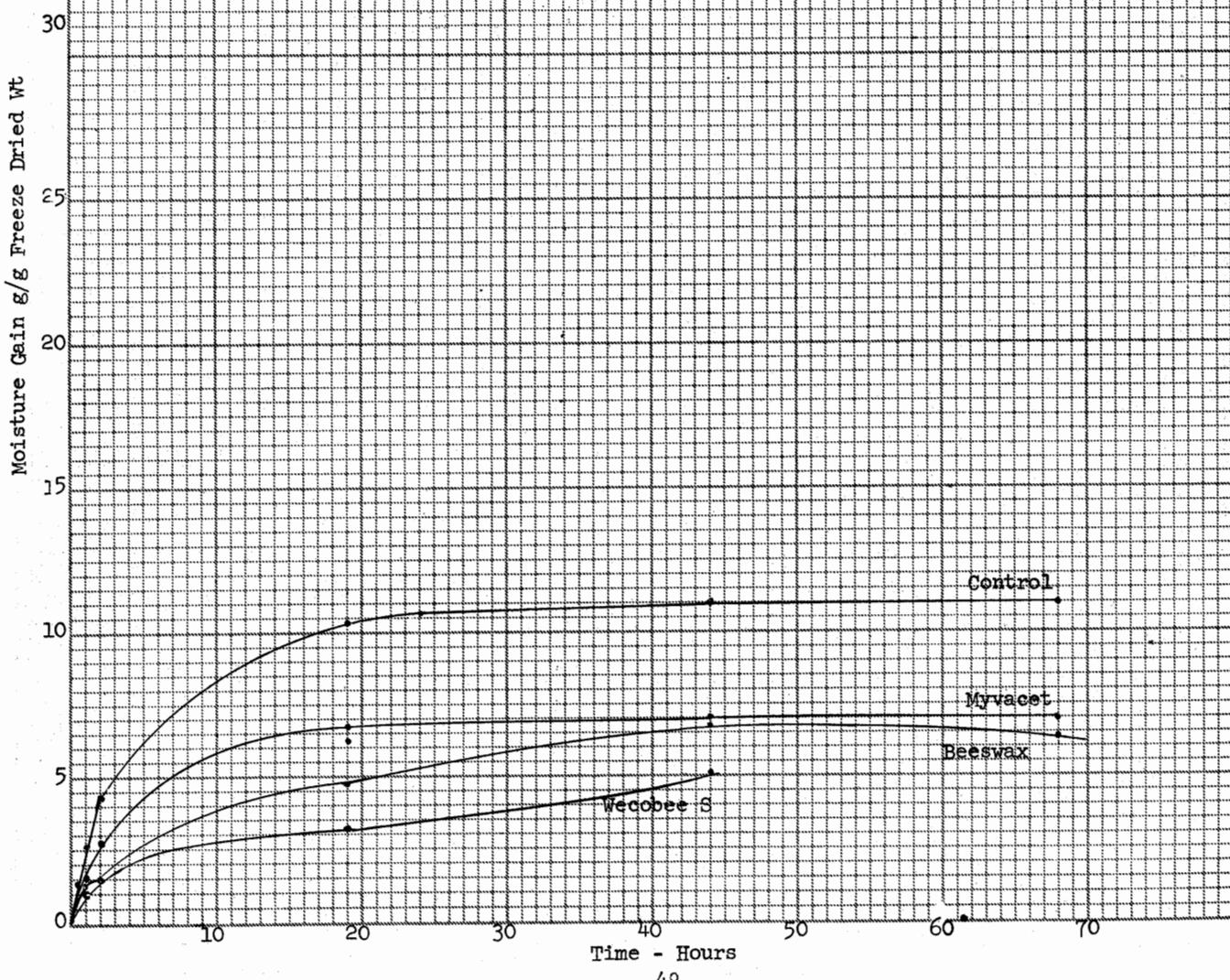


FIGURE 1
MOISTURE GAIN BY FREEZE DRIED BEEF COATED IN THE TERMINAL STAGE
OF FREEZE DRYING AND STORED AT 75% RH, 23°C.

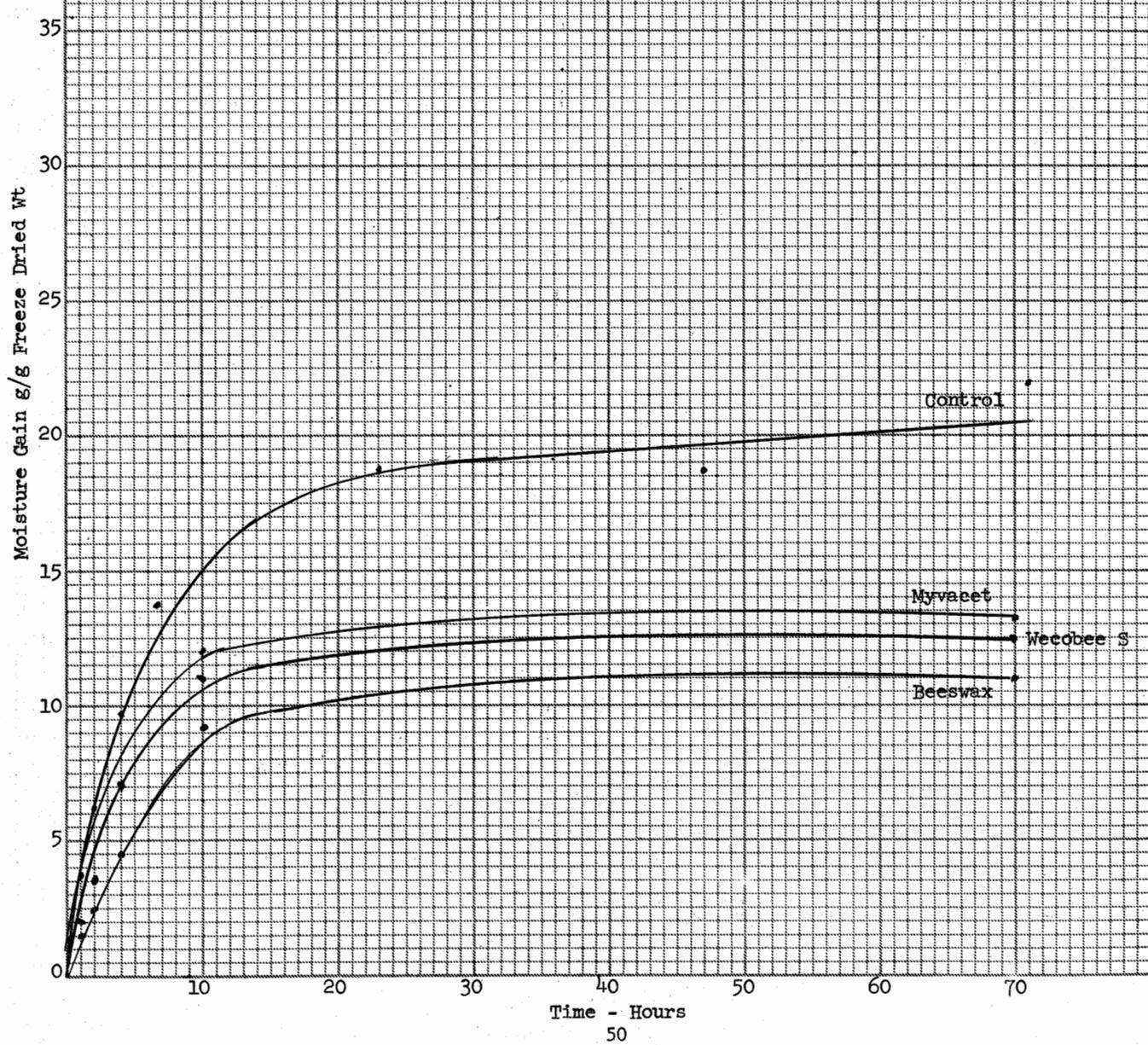


FIGURE 8
MOISTURE GAIN BY FREEZE DRIED BEEF COATED IN THE
TERMINAL STAGE OF FREEZE DRYING AND STORED AT 98% RH, 23°C.

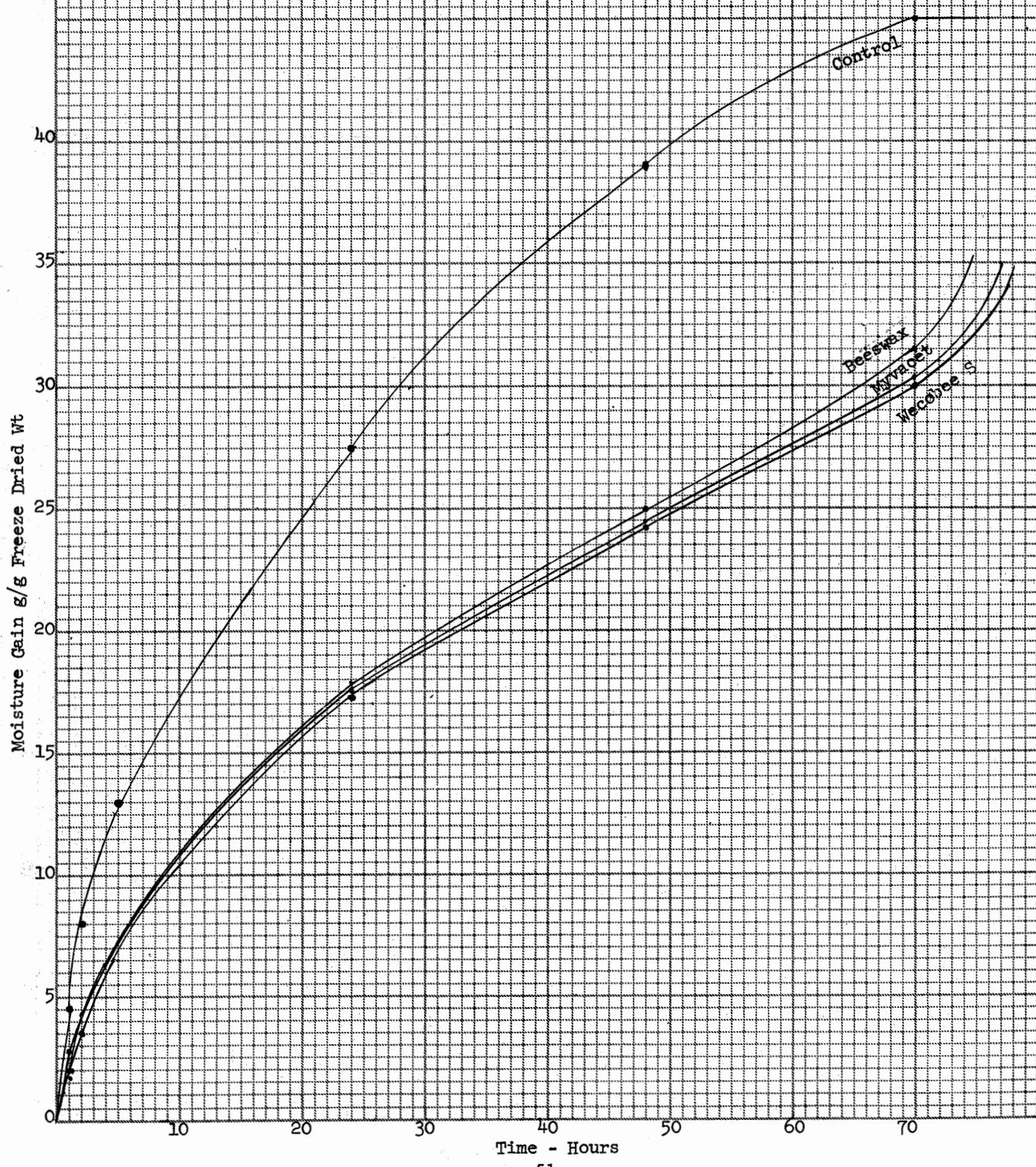
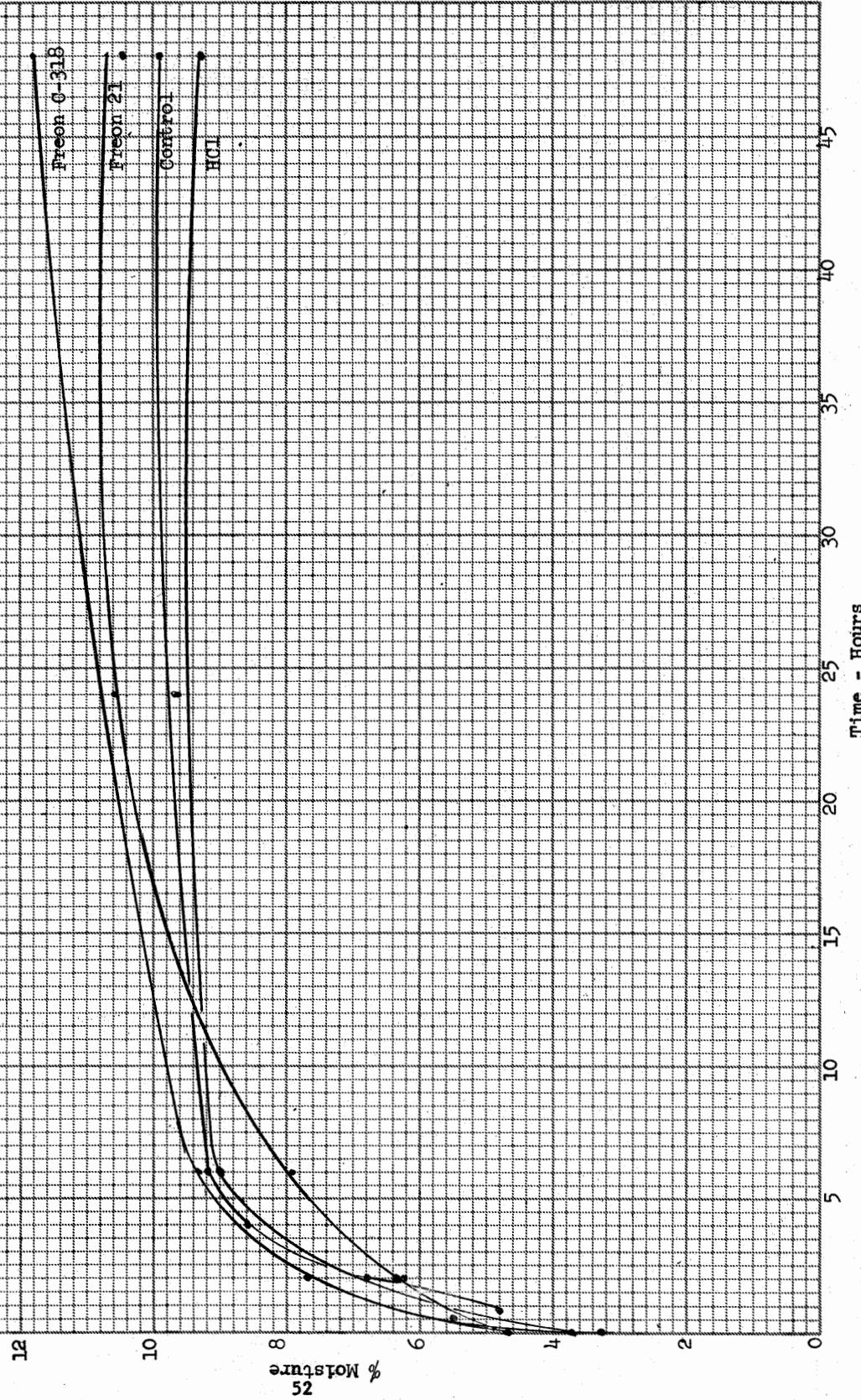


FIGURE 9
MOISTURE SORPTION BY GAS-TREATED FROZEN COOKED CHICKEN CHIPS



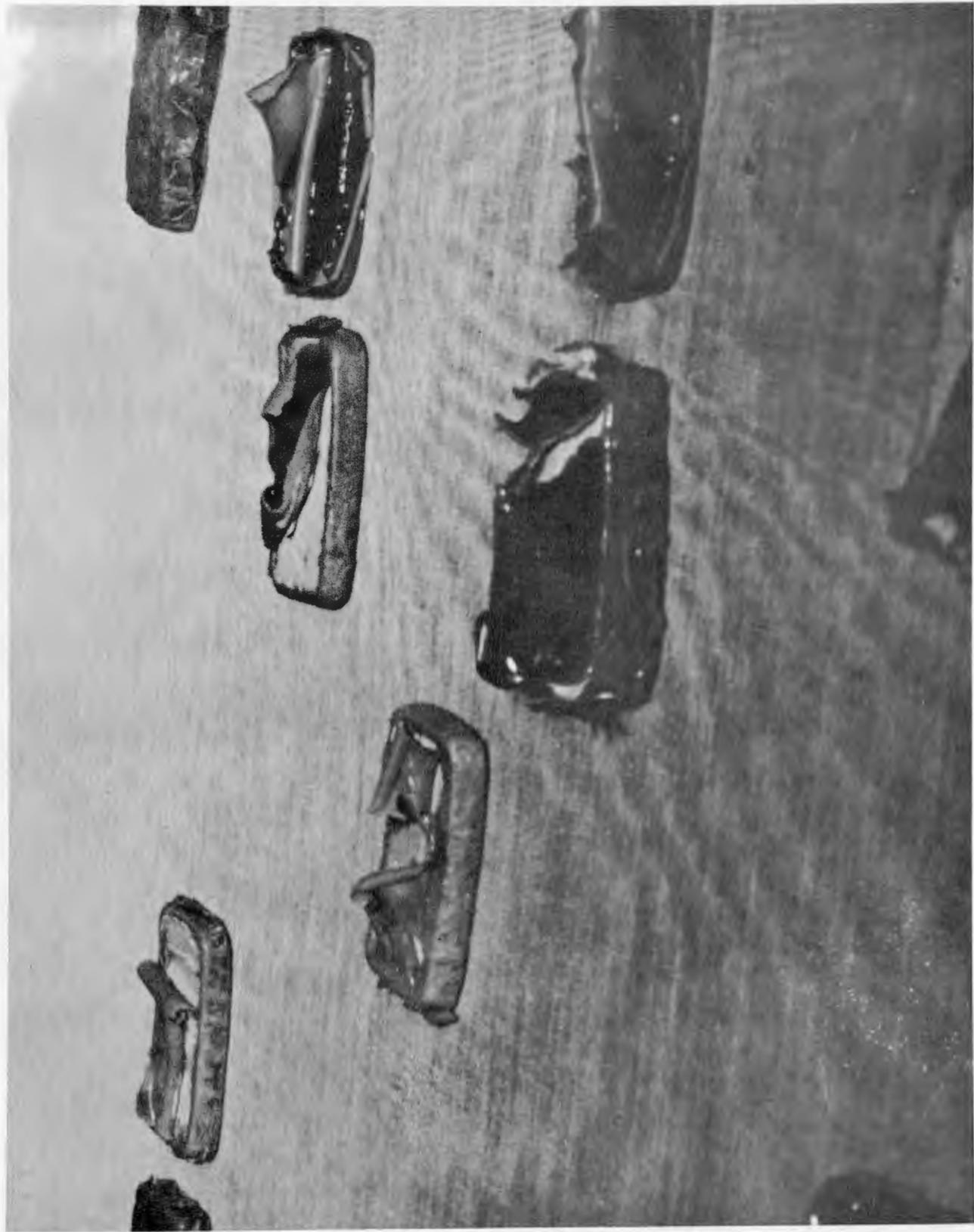


FIGURE 10

PEELING OF COATED FOOD BARS UNDER CYCLED TEMPERATURES AND HUMIDITY CONDITIONS

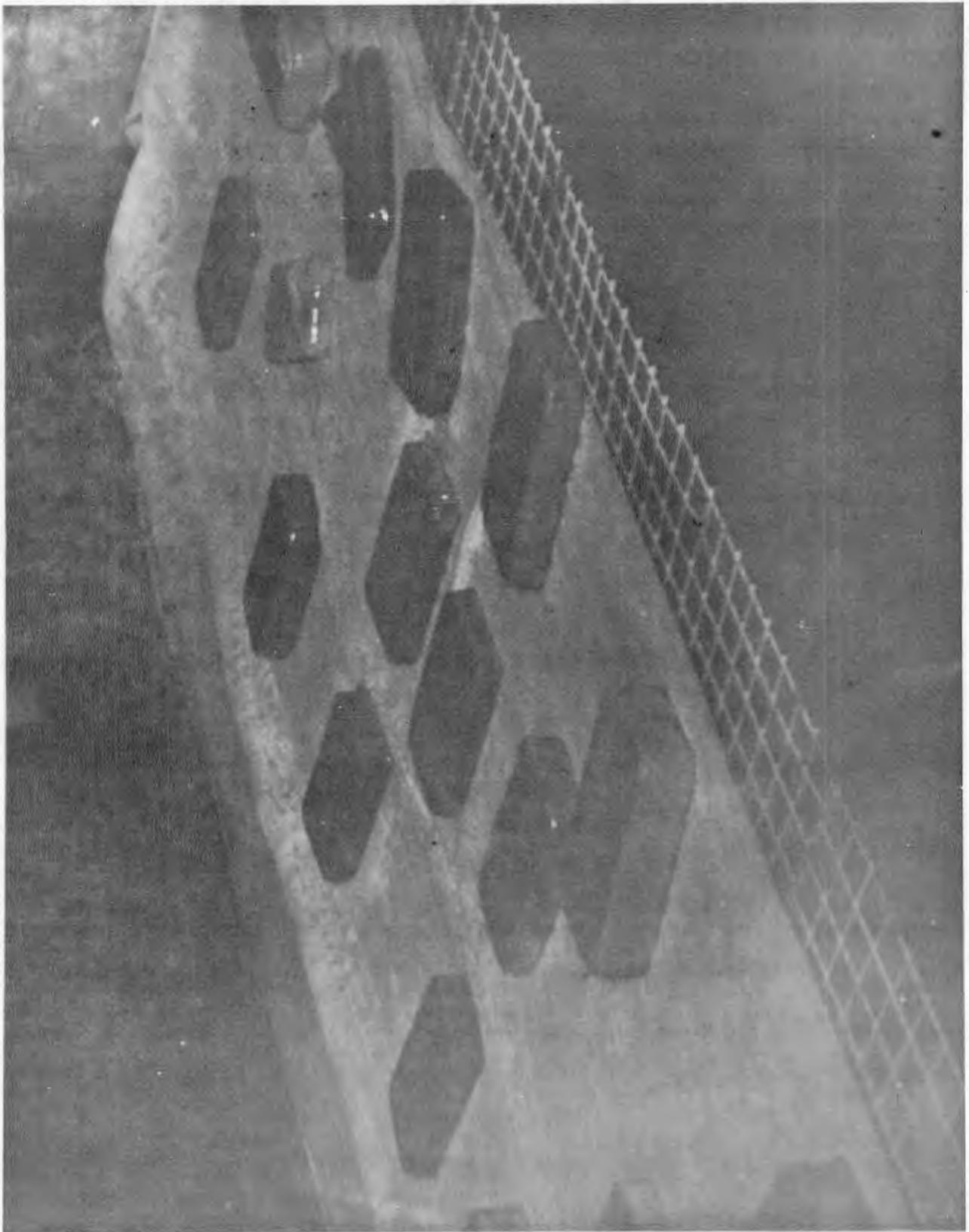


FIGURE 11
COATED FOOD BARS

DISTRIBUTION

Copies

1 - Commanding General
US Army Weapons Command
ATTN: AMSWE-RDR
Rock Island, Illinois 61200

1 - Commanding General
USA Test & Evaluation Command
ATTN: AMSTE-TAA
Aberdeen Proving Ground
Maryland 21005

1 - Commanding General
US Army Nuclear Defense
Laboratory
Army Chemical Center
Maryland 21005

1 - Commanding General
United States Continental
Army Command
ATTN: DCSLOG, Maintenance
Division
Fort Monroe, Virginia 23351

1 - Commanding General
USA Combat Developments Command
ATTN: CDCMR-O
Fort Belvoir, Virginia 22060

1 - Commanding General
US Army Mobility Command
ATTN: AMSMO-RR
Warren, Michigan 48089

1 - Commanding Officer
US Army Polar Research and
Development Center
Fort Belvoir, Virginia 22060

1 - Commanding General
US Army Edgewood Arsenal
ATTN: Directorate of Commodity
Management
Edgewood Arsenal, Maryland 21010

1 - Commanding General
US Army Electronics
Research and Development
Laboratories
ATTN: Tech Info Div
Fort Monmouth, NJ 07703

1 - Commanding Officer
US Army Materials Research
Agency
ATTN: Technical Library
Watertown, Massachusetts 02172

1 - Commanding Officer
USACDC Nuclear Group
Fort Bliss, Texas 79916

1 - Commanding Officer
US Army Combat Developments
Command
Combat Service Support Group
Fort Lee, Virginia 23801

1 - Commanding Officer
US Army Combat Developments
Command
ATTN: CDCQMA-F
Fort Lee, Virginia 23801

2 - Commanding Officer
Cold Weather & Mountain
Indoctrination School
Fort Greely, Alaska

1 - Commanding Officer
US Army Research Office-
Durham
ATTN: CRD-AA-IP
Box CM, Duke Station
Durham, North Carolina 27706

1 - Commanding Officer
US Army Cold Regions Research
& Engineering Laboratories
Hanover, New Hampshire 03755

Copies

1 - Commanding Officer
US Army Human Engineering
Laboratories
Aberdeen Proving Ground
Maryland 21005

1 - Commanding Officer
US Army Coating & Chem Labs
Aberdeen Proving Ground
Maryland 21005

1 - Directorate of Science and
Technology
AFRSTD
DCS/R&D
Hq., United States Air Force
Washington, D. C. 20330

1 - Director
US Army Engineer Research and
Development Laboratories
ATTN: Technical Document Ctr
Fort Belvoir, Virginia 22060

1 - Director
Air University Library
ATTN: AUL3T-7575
Maxwell AFB, Alabama 36112

1 - Director
Biological Sciences Division
Office of Naval Research
Department of the Navy
Washington, D. C. 20360

1 - Director, Library
US Army War College
Carlisle Barracks
Pennsylvania 17013

1 - Director
Aerospace Crew Equip Lab
Naval Air Engr Center
Philadelphia, Pa 19112

1 - Director
Marine Corps Landing Force
Development Center
Marine Corps Schools
ATTN: Ground Combat Division
Quantico, Virginia 22134

1 - Director
Engineering & Ind Svcs
ATTN: Directorate of
Chemical Engineer
Edgewood Arsenal, Md 21010

1 - Director
Army Technical Information
US Army Research Office
OCRD, Room 209A
Arlington, Virginia 22200

2 - Commandant
US Army Quartermaster School
ATTN: Quartermaster Library
Fort Lee, Virginia 23801

1 - Commandant
USA Armor School
ATTN: Ch, Pol & Tng Lit Div
Fort Knox, Ky 40121

2 - Commandant
US Army Infantry School
ATTN: AJIIS-A
Fort Benning, Georgia 31905

1 - Commandant of the Marine Corps
Headquarters Marine Corps
CODE A04D
Washington, D. C.

1 - Commander
US Naval Ordnance Test Station
ATTN: Code 12
China Lake, California 93557

1 - Commander
US Army Chemical Research &
Development Lab
ATTN: Technical Library
Edgewood Arsenal, Md 21010

1 - Commander
US Army Biological Labs
ATTN: Technical Library
Fort Detrick
Frederick, Maryland 21701

Copies

1 - President
Hq, US Army Artillery Board
Fort Sill, Oklahoma 73504

2 - Commanding Officer
US Army Arctic Test Center
APO Seattle 98733

1 - President
US Army Aviation Test Board
Fort Rucker, Alabama 36362

1 - President
US Army Infantry Board
Fort Benning, Georgia 31905

1 - President
US Army Armor Board
Fort Knox, Kentucky 40121

1 - The Army Library
ATTN: Procurement Section
Room 1A522, The Pentagon
Washington, D. C. 20301

1 - Library
US Weather Bureau
Washington, D. C. 20235

1 - Air Force Cambridge Research Labs
Laurence G. Hanscom Field
ATTN: CRMCLR, Res Library, Stop 29
Bedford, Massachusetts 01731

1 - US Army Aviation School Library
Bldg 5313
Fort Rucker, Alabama

2 - Chief, Status and Support Branch
Maintenance Readiness Division
US Army Supply & Maintenance Cmd
Washington, D. C. 20315

1 - US Army Ballistic Research Lab
ATTN: AMXBR-TC, Mr. B. F. Armendt
Aberdeen Proving Ground
Maryland 21005

1 - United States Dept of
Agriculture
Division of Acquisitions
National Agricultural Library
Washington, D. C. 20250

1 - Library
US Army Airborne, Electronics &
Special Warfare Board
Fort Bragg, North Carolina 28307

1 - US Atomic Energy Commission
Reports Section, Hqs Library
Mail Station G-017
Division of Technical Info
Washington, D. C. 20545

1 - ACOFS, G3
Hq, US Army Combat Dev Cmd
Experimentation Center
Fort Ord, California 93941

2 - Redstone Scientific Information
Center
US Army Missile Command
ATTN: Ch, Documents Section
Redstone Arsenal, Alabama 35808

1 - US Army Special Warfare School
ATTN: Asst Secretary
Director of Instruction
Fort Bragg, North Carolina 28307

3 - US Atomic Energy Commission
Division of Technical Information
Extension
PO Box 62
Oak Ridge, Tennessee 37831

1 - US Naval Research Laboratory
Code 6140
Washington, D. C. 20390

1 - Chief, Bureau of Ships
Room 2510, Main Navy
Code 364A4
18th & Constitution Ave, N W
Washington, D. C. 20001

Copies

1 - Chief, Programs & Policy Office
Directorate of Tech Operations
DCTSC
2800 South 20th Street
Philadelphia, Pa 19101

1 - US Naval Applied Science Lab
Technical Library
Bldg 291, Code 9832
Naval Base
Brooklyn, New York 11251

1 - US Army Command & Gen Staff
College
Library Division
Fort Leavenworth, Kansas 66027

1 - Exchange and Gift Division
Library of Congress
Washington, D. C. 20540

2 - Chief, Supply Division
Logistics Services
Hq, Fort Monmouth
Vail Hall, Bldg 1150
Ft Monmouth, NJ 07703

1 - Department of the Navy
Special Projects Office
Washington, D. C. 20360

1 - Arctic AEROMED Laboratory
ATTN: Librarian
APO 731, Seattle, Washington

1 - US Army Materiel Command
Research Division AMCRD-RL
R&D Directorate
Bldg T-7
Washington, D. C. 20315

1 - US Army Engineering Research
Development Laboratories
ATTN: STINFO Branch
Fort Belvoir, Virginia 22060

1 - Library
US Naval Supply Research and
Development Facility
Naval Supply Center
Bayonne, NJ 07002

1 - Reference Center Library
The Institute for Cooperative
Research
Eglin Facility
PO Box 1867
Eglin Air Force Base, Florida

1 - Mr. Gerald Chaikin
US Army Missile Command
ATTN: AMSMI-PC
Redstone Arsenal, Alabama 35808

1 - National Research Council
University of Rhode Island
Kingston, Rhode Island 02881

1 - Senior Standardization
Representative
US Army Standardization Group,
Canada
ATTN: US Army Stdzn Rep (QMAE)
c/o Directorate of Interservice
Development
138 Queen Street
Ottawa, Ontario, Canada

1 - NASA Scientific and Technical
Information Facility
ATTN: Acquisitions Branch
(S-AK/DL)
PO Box 33
College Park, Maryland 20740

1 - Library
Southern Utilization R&D Div
Agricultural Research Service
US Department of Agriculture
PO Box 19687
New Orleans, Louisiana 70119

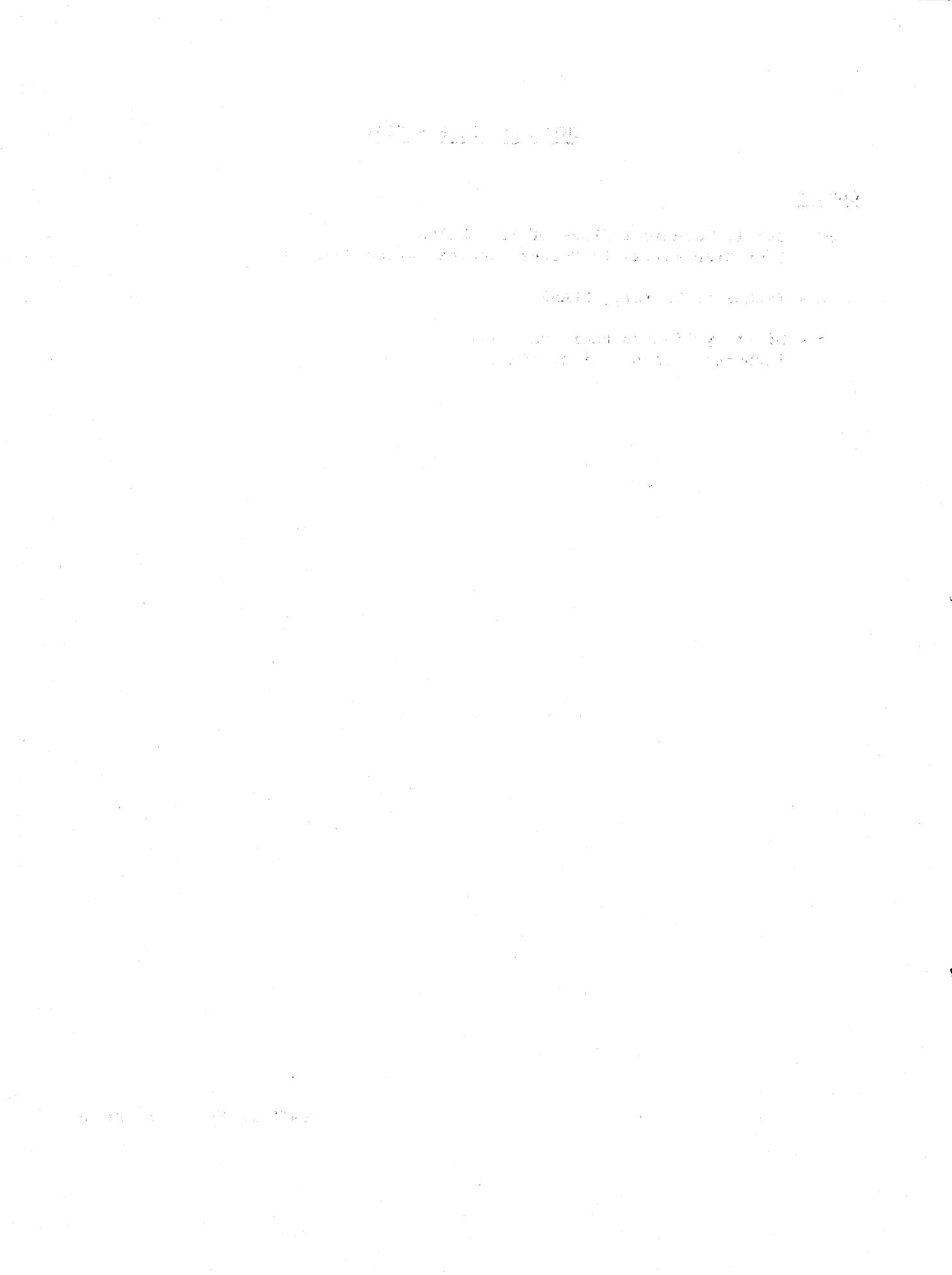
1 - USA NLABS Liaison Office
ASDL-8
Wright-Patterson AFB, Ohio

INTERNAL DISTRIBUTION

Copies

- 20 - Chief, Technical Plans Office, NLABS
(for transmittal to Defense Documentation Center)
- 2 - Technical Library, NLABS
- 5 - Military Liaison Representative
Technical Plans Office, NLABS

DATE PRINTED: 1 Apr 66



Unclassified

Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Archer Daniels Midland Company Minneapolis, Minnesota	2a. REPORT SECURITY CLASSIFICATION Unclassified
	2b. GROUP

3. REPORT TITLE

EDIBLE COATINGS FOR DRIED AND COMPAKTED FOODS: PART II

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Final: Part II

5. AUTHOR(S) (Last name, first name, initial)

Cole, Morton S.

6. REPORT DATE

May 1966

7a. TOTAL NO. OF PAGES

54

7b. NO. OF REFS

9

8a. CONTRACT OR GRANT NO.

DA19-129-AMC-102(N)

9a. ORIGINATOR'S REPORT NUMBER(S)

b. PROJECT NO.

7X84-06-033

9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

66-38-FD FD-48

10. AVAILABILITY/LIMITATION NOTICES

Distribution of this document is unlimited.

Release to CFSTI and DDC is authorized.

11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY

**U. S. Army Natick Laboratories
Natick, Massachusetts 01760**

13. ABSTRACT

Edible thermoplastic coatings were applied as moisture-oxygen barriers to dry bars representing a wide range of food compositions. A hot-melt curtain coating technique appears commercially feasible for all foods examined; however, coatings so applied failed to withstand storage at a temperature cycling between -18 and + 40°C.

Unclassified

Security Classification

14.

KEY WORDS

Applying
Coatings
Food
Food bars
Compressed
Spraying
Dipping
Fusion (Melting)

		LINK A		LINK B		LINK C	
ROLE	WT	ROLE	WT	ROLE	WT	ROLE	WT
	8						
	1						
	1						
	1,2						
	0						
	10						
	10						
	10						

INSTRUCTIONS

1. ORIGINATING ACTIVITY: Enter the name and address of the contractor, subcontractor, grantees, Department of Defense activity or other organization (corporate author) issuing the report.

2a. REPORT SECURITY CLASSIFICATION: Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.

2b. GROUP: Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.

3. REPORT TITLE: Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parenthesis immediately following the title.

4. DESCRIPTIVE NOTES: If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.

5. AUTHOR(S): Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.

6. REPORT DATE: Enter the date of the report as day, month, year; or month, year. If more than one date appears on the report, use date of publication.

7a. TOTAL NUMBER OF PAGES: The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.

7b. NUMBER OF REFERENCES: Enter the total number of references cited in the report.

8a. CONTRACT OR GRANT NUMBER: If appropriate, enter the applicable number of the contract or grant under which the report was written.

8b, 8c, & 8d. PROJECT NUMBER: Enter the appropriate military department identification, such as project number, subproject number, system numbers; task number, etc.

9a. ORIGINATOR'S REPORT NUMBER(S): Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.

9b. OTHER REPORT NUMBER(S): If the report has been assigned any other report numbers (either by the originator or by the sponsor), also enter this number(s).

10. AVAILABILITY/LIMITATION NOTICES: Enter any limitations on further dissemination of the report, other than those imposed by security classification, using standard statements such as:

- (1) "Qualified requesters may obtain copies of this report from DDC."
- (2) "Foreign announcement and dissemination of this report by DDC is not authorized."
- (3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through _____."
- (4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through _____."
- (5) "All distribution of this report is controlled. Qualified DDC users shall request through _____."

If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.

11. SUPPLEMENTARY NOTES: Use for additional explanatory notes.

12. SPONSORING MILITARY ACTIVITY: Enter the name of the departmental project office or laboratory sponsoring (paying for) the research and development. Include address.

13. ABSTRACT: Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as (TS), (S), (C), or (U).

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. KEY WORDS: Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, rules, and weights is optional.

Unclassified

Security Classification